



**2018 Results for Per- and Polyfluoroalkyl Substances  
(PFAS) Analyses Performed by United States  
Environmental Protection Agency's Office of Research  
and Development for Samples Collected in Southern  
New Hampshire**

*April 2019*

# **2018 Results for Per- and Polyfluoroalkyl Substances (PFAS) Analyses Performed by United States Environmental Protection Agency's Office of Research and Development for Samples Collected in Southern New Hampshire**

## **2018 Summary Report**

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## 1. Introduction

In 2017, the New Hampshire Department of Environmental Services (NHDES) requested assistance from the United States Environmental Protection Agency's Office of Research and Development (EPA ORD) to conduct analyses of various samples for the potential existence of per- and polyfluoroalkyl substances (PFAS) (Appendix A-1 and A-3). The type of samples identified by NHDES for analysis included industrial coating formulations, air emissions, groundwater, surface water, soil and leachate. Specifically, NHDES was interested in samples taken near sites where there was the potential for air emissions associated with processes that historically and currently use PFAS-containing raw materials. The release of PFAS compounds into the environment from these sites has contaminated soil and water, including groundwater used as drinking water for tens of thousands of people in the state.

Assistance from EPA ORD was requested in order to address several technical barriers that included: 1) commercial laboratory analytical limitations for handling more complex sample matrices; 2) unknown nature of the compounds because they are either proprietary, manufacturing byproducts contained in raw materials, or degradation compounds; and 3) lack of expertise and experience associated with advanced fluorochemistry and fate and transport properties of this class of compounds. Specifically, EPA ORD has unique capabilities to conduct "non-targeted" screening, whereby a search is conducted of a broader spectrum of PFAS that may be present in a sample, including both known and unknown compounds; this type of analysis is not available through a commercial laboratory.

NHDES has several objectives for this on-going partnership with EPA to evaluate PFAS in samples collected from New Hampshire:

- 1) Understand the multimedia environmental distribution of fluorinated compounds originating from PFAS emissions to air;
- 2) Differentiate sources of contamination when and where there is the potential for multiple PFAS sources by identifying a signature distribution of compounds for different sources of PFAS;
- 3) Assess if conditions warrant the installation of treatment systems for air emissions to proactively prevent the contamination of the environment and drinking water with newer alternative chemicals or precursor compounds;
- 4) Ensure any potential air pollution control equipment systems that may be required to be installed are designed to remove not only perfluorooctanoic acid (PFOA) and perfluorooctane sulfonic acid (PFOS), but a broader array of PFAS compounds and associated break-down products, some which could be precursors to the formation of PFOA and PFOS. This information is critical for ensuring any air pollution control equipment is properly designed for potential PFAS emissions and that remediation

systems removing PFOA and PFOS do not unknowingly redistribute other PFAS contaminants;

- 5) Ensure drinking water treatment systems and remediation systems being designed to remove PFOA and PFOS are also able to remove other PFAS compounds and associated break-down products, some which could be precursors to the formation of PFOA and PFOS. This information is critical for ensuring drinking water is appropriately treated and that remediation systems removing PFOA and PFOS do not unknowingly redistribute other PFAS contaminants; and
- 6) Prioritize which contaminant(s) need a risk assessment based on what is actually being measured in the environment, including drinking water.

In 2018, the EPA produced four reports that document the results of analyses conducted to date as part of the ongoing partnership with NHDES. These reports are the first in what is expected to be a series of reporting by the EPA on the results of New Hampshire sample analyses. In many respects, the analytical work conducted by EPA ORD is at the forefront of the emerging science of PFAS. NHDES is pleased to be working closely with EPA ORD on this contamination issue that is of such vital concern to the citizens of New Hampshire.

The EPA data reports are intended to provide a simple representation and summary of the analysis results. Therefore, the description of methods and quality assurance are brief and high-level. Additional reports and/or publications are being developed that will include a more detailed description of methods, quality assurance analyses, and statistical/geospatial interpretation of the data. As study partners/collaborators, it is anticipated that NHDES and EPA Region 1 scientists will assist in these additional reports and publications.

## **2. Project Overview**

NHDES and EPA jointly developed two work plans for the sampling and analysis related to the investigation into releases of PFAS compounds to the air from industrial facilities in NH. Those plans are:

*Work Plan #1: Southern New Hampshire Sample Collection Plan for Non-Targeted Per- and Poly-fluorinated Compounds Analyses* dated August 30, 2017. (Appendix A-2)

This plan guided the collection and analyses of samples from several different environmental media including groundwater, surface water, and soil as well as industrial samples of raw materials used in dispersions at two facilities and solid material (referred to as char) that accumulates inside the air emission stacks.

*Work Plan #2: Saint-Gobain Performance Plastics Air Sample Collection Plan for Non-Targeted Per- and Polyfluoroalkyl Substances Analysis* dated April 18, 2018. (Appendix A-4)

This plan guided the collection and analyses of samples from process feedstocks, pilot-scale air pollution control equipment, and air emissions from three towers during a multiday stack testing event in April and May 2018 at Saint-Gobain Performance Plastics (SGPP).

### **3. Sample Collection**

#### *3.1 Work Plan #1:*

NHDES staff collected samples of groundwater, surface water, soil, raw materials used in dispersions, and char in general accordance with Work Plan #1, as described in the sections below. Samples were then submitted to EPA ORD for analysis. For logistical reasons, some of the samples proposed in the Work Plan were not able to be collected (e.g., dust was not able to be sampled due to lack of sufficient sample volume).

##### *3.1.1 Groundwater*

A total of 20 groundwater samples (including three duplicate samples) were collected and submitted to EPA ORD (Figure 1, Table 1). Groundwater sources sampled for this study included monitoring wells and private wells no longer used for drinking. Wells were selected for inclusion in this study based on results of prior PFAS analyses so that a range of PFAS concentrations could be evaluated, including highly contaminated monitoring wells close to the facility, wells with PFOA concentrations between 0.05 and 0.10 ng/L, and wells with PFOA concentrations exceeding 0.20 ng/L.

##### *3.1.2 Surface Water*

Surface water samples (including one duplicate sample) were collected from five different locations and submitted to EPA ORD (Figure 1, Table 1). Samples were collected from the Merrimack River (EPAORD 002 and EPAORD 003) and Dumpling Brook (EPAORD 001, EPAORD 003, and duplicate sample EPAORD 004). Discharge from the stormwater system that drains the SGPP facility was sampled at the Outfall (EPAORD 006) during dry conditions when the water discharging from the system is likely due to groundwater infiltration.

##### *3.1.3 Soil*

Three soil samples were collected from different depths at a single boring location near the SGPP facility (Figure 1, Table 1). NHDES staff advanced the boring to a total depth of six feet below the ground surface using a stainless steel hand auger. The three intervals sampled for this study included the surface soil (EPAORDS1 or NHEPAORD-S1), an interval two to four feet below ground (EPAORDS2 or NHEPAORD-S2), and the interval from four to six feet below ground (EPAORDS3 or NHEPAORD-S3).

### *3.1.4 Char Material from Tower Stacks*

NHDES staff collected three samples of solid material (char) that coats the interior of the stacks of the textile coating towers. The three towers were chosen because they have the potential to represent three different operational situations (Figure 1, Table 1):

- 1) The MA Tower has been in operation since 1994. The ductwork was replaced and the oven and ancillary process components were cleaned in 2016. Therefore, the solid material (Sample ID: MA Tower Char or NHCharMA) that was collected from this stack will most likely represent “new” dispersions used since 2016.
- 2) The MS Tower has been in operation since 2002. The solid material (Sample ID: MS Tower Char or NHCharMS) that was collected from this stack may potentially contain residue components from pre-2006 PFOA-based dispersions AND “new” dispersions that have been used since 2006.
- 3) The QX Tower has been in operation since 1989. According to historical stack testing results, the QX Tower receives the highest load of emissions, and solid material (Sample ID: QX Tower Char or NHCharQX) that was collected from this stack would likely be associated with emissions that occurred while PFOA-based dispersions were in use and emissions that occurred after the use of PFOA had been phased out.

### *3.1.5 Raw Materials used in Dispersions*

NHDES staff collected a total of thirteen raw material samples (Sample IDs: 1 – 13) from the two textile coating facilities. According to company representatives and records, these samples represent all of the raw materials containing fluorinated compounds currently stored or in use at the two facilities.

## *3.2 Work Plan #2:*

In addition to and in conjunction with Work Plan #2, NHDES requested that SGPP conduct air emission stack testing of three towers in order to collect the samples for analysis by EPA ORD. The stack tests were conducted April 26 – May 2, 2018 by SGPP and their consultants while NHDES personnel were present to observe the process operations, sampling, and stack testing methodology and also to facilitate transmission of samples to EPA ORD. The stack tests were conducted in accordance with the approved stack test plan submitted by SGPP and dated April 11, 2018. (Appendix A-5). Stack tests were conducted on the MA, MS and QX Towers. Samples were taken from the QX Tower at the inlet and outlet of a pilot-scale control device to evaluate its effectiveness at removal of PFAS compounds. Table 2 is a summary of all of the samples submitted to EPA ORD collected as part of Work Plan #2.



### *3.2.1 Air Emission Samples*

During each of the stack tests, a total of three runs were completed. During each run, a sample train comprised of seven segments was deployed. In total, 84 air emission samples were collected. These samples were taken by stack testing personnel and sent directly to EPA ORD for analysis. (Sample IDs: 500 – 520, 600 – 620, 700 – 720, 800 – 820)

### *3.2.2 SUMMA Canister Samples*

There were 18 SUMMA canister samples collected including two background samples, four ambient samples and 12 stack test runs. These samples were collected by NHDES personnel and sent directly to EPA ORD for analysis. (Sample IDs: 2 backgrounds, 709, 721, 176, A378, 321, 2045, 793, 005, 755, 751, 262, RK9, 068, 700, 744, 794)

### *3.2.3 Dip Tank Coatings*

Eleven total samples of actual coating utilized during each stack test were taken by stack testing personnel from the dip tank(s) for each tower and sent directly to EPA ORD for analysis. (Sample IDs: 521, 522, 523, 621, 622, 623, 721, 722, 725, 726, 729)

### *3.2.4 Char Material from Tower Stacks*

NHDES staff collected three samples of solid material (char) that coats the interior of the stacks of the three textile coating towers that were stack tested. (Sample IDs: MA, MS QX)

### *3.2.5 Process Water from Pilot-Scale Air Pollution Control Equipment*

Four samples of the process water that was used in the pilot scale air pollution control device were sampled by stack testing personnel and sent directly to EPA ORD for analysis. (Sample IDs: 733, 734, 735, 736)

## **4 EPA ORD Reports**

EPA ORD submitted four reports to NHDES in 2018 that convey results of analyses performed on the various samples from New Hampshire (Table 1 and Table 2). These reports represent the first in a series of data that will be presented to NHDES by EPA ORD; additional reports will be provided as EPA ORD finishes their analyses. Each report is included in its entirety in Appendices A-6 through A-9. The exhibit below summarizes the types of results provided in each report.

## Exhibit 1

Report Number	Date	Sample Media	Type of Analyses
ORD Report #1	April 4, 2018	Char and Soil	Targeted PFCAs
ORD Report #2	July 24, 2018	Char and Soil	Non-Targeted PFAS
ORD Report #3	October 4, 2018	Groundwater and Surface Water	Targeted PFAS
ORD Report #4	October 4, 2018	Stack Emissions SUMMA Canister Samples Only	Non-Targeted PFAS and Volatile Organic Hazardous Air Pollutants

### 4.1 ORD Report #1

ORD Report #1 contains the results of targeted perfluorocarboxylate<sup>1</sup> (PFCA) concentrations for the two types of solid-matrix samples analyzed at the EPA ORD laboratory located in Athens, Georgia (Appendix A-6). Three samples each of char and soil were analyzed for thirteen PFCAs using liquid chromatography / mass spectrometry. Internal standards of each compound were used to quantify the laboratory results in terms of micrograms per gram ( $\mu\text{g/g}$  or parts per million (ppm)) for char and picograms per gram ( $\text{pg/g}$  or parts per trillion (ppt)) for soil. Key results of the report include:

- Overall PFCA concentrations were greater in the char samples (reported in ppm) compared to the soil samples (reported in ppt).
- Chain lengths of the PFCAs detected in char ranged from four carbons (C4) to 18 carbons (C18); whereas, the longest chain PFCA detected in soil was C11.
- PFOA was the PFCA with the highest concentration in each of the six samples.
- PFCA concentrations generally decreased with depth in the three samples from the soil boring.

### 4.2 ORD Report #2

ORD Report #2 contains the results of non-targeted PFAS concentrations for the two types of solid-matrix samples analyzed at the EPA ORD laboratory located in Athens, Georgia (Appendix A-7). The same samples of char and soil described in ORD Report #1 were analyzed for non-targeted PFAS using liquid chromatography / mass spectrometry. Known standards are not available for the analysis of the non-targeted compounds, therefore, there is more uncertainty in terms of identification of the compounds and estimation of concentrations. The results reported in units of  $\mu\text{g/g}$  for char and  $\text{pg/g}$  for soil, are considered to be estimated values. In other words, the results are “semi-quantitative, likely within an order of magnitude of the actual value” and have a greater level of uncertainty relative to results of analyses performed with known standards. Key results of the report include:

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<sup>1</sup> Perfluorocarboxylates are reported as perfluorocarboxylic acids.

- Two types of PFAS were detected that have not been previously identified in environmental samples and include a polyfluoroalkyl carboxylic acid series and a polyfluorinated sulfonic acid (PFSA) series, each of which has a hydrogen atom substituted for a fluorine atom. The hydrogenated PFCA and PFSA series are referred to as HPFCA and HPFSA, respectively, in Report #2. Actual concentration values for HPFSA were not presented because they occurred at lower concentrations relative to HPFCA.
- The number of carbon atoms in the HPFCAs detected in the samples ranged from C6 to C20 and from C4 to C18 for the HPFSAs.
- Similar to PFCA results in Report #1, HPFCA concentrations were orders of magnitude greater in the char samples (reported in ppm) compared to the soil samples (reported in ppt).
- HPFSA concentrations were greater than HPFCA by a factor of 10 and 1.2 for C6 and C8, respectively in the char samples. For the other carbon chain lengths, HPFCA was present at greater concentrations than HPFSA.
- The surface soil sample (EPAORDS1 or NHEPAORD-S1) contained the greatest concentrations of HPFCA compared to deeper soil samples.

#### 4.3 ORD Report #3

ORD Report #3 contains the results of targeted PFAS concentrations for groundwater and surface water samples analyzed at the EPA ORD laboratory located in Research Triangle Park, North Carolina (Appendix A-8). Twenty-five aqueous samples were analyzed for 11 PFAS using liquid chromatography / mass spectrometry. Standards of each compound were used to quantify the laboratory results in terms of nanograms per liter (ng/L). Key results of the report include:

- PFOA was detected in 22 of 25 samples. In all samples where it was detected, PFOA had the greatest concentration of all of the other PFAS detected.
- PFOS was not detected in any of the groundwater samples; however, it was detected in four of the five surface water samples and the sample from the stormwater outfall.
- Perfluoro(2-methyl-3-oxahexanoic) acid, also known as GenX, a compound that is associated with a newer generation of PFAS that replaced PFOA, was detected in one groundwater sample from a domestic well (EPAORD 016) above the limit of quantification (LOQ) at a concentration of 35.4 ng/L. To NHDES' knowledge, this represents the first detection of GenX in groundwater from a domestic well out of hundreds of samples from other domestic wells that have been tested for this compound by commercial laboratories. In consultation with EPA ORD staff, it is suggested that this sample be reanalyzed for conformational analysis of this unique finding and rule out laboratory contamination.

#### 4.4 ORD Report #4

ORD Report #4 contains the results of non-targeted PFAS and volatile organic hazardous air pollutants (HAPs) analyses for the gases collected in SUMMA canisters during the stack test in April and May, 2018. Analyses were conducted at the EPA ORD laboratory located in Research Triangle Park, North Carolina (Appendix A-9). Due to this being the first time SUMMA canister sampling for PFAS compounds has been performed on stack emissions, the high resolution chemical ionization mass spectrometry (CIMS) analyses for the non-targeted PFAS are limited to tentative identifications, rather than quantification. The more conventional TO-15 analysis<sup>2</sup> using unit mass resolution (low resolution) gas chromatography-mass spectrometry (GC-MS) can be understood as a targeted analysis of gas phase volatile organic hazardous air pollutants (HAPs). Key results of the report include:

- The CIMS-based non-targeted analysis tentatively identified twelve (12) PFAS compounds in the SUMMA canisters.
- The GC-MS targeted selective ion monitoring (SIM) analysis of SUMMA canisters identified 27, 42 and 38 non-PFAS compounds in the samples from the MS, MA and QX towers, respectively.
- Up to 118 gas phase compounds were observed across all nine samples using the TO-15 (plus photochemical assessment monitoring station compounds) method.
- Across all nine SUMMA canisters representing non-controlled stack emissions, the following compounds were observed in all canisters: propylene, propane, chloromethane, isobutene, 1-butene, ethanol, acrolein, acetone, iso-pentane, isopropyl alcohol, 1-pentene, isoprene, vinyl acetate, 2-butanone, 1-hexene, tetrahydrofuran, 2,4-dimethylpentane, benzene, 4-methyl-2-pentanone, toluene, and dodecane.

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<sup>2</sup> TO-15 is an EPA approved method for determination of volatile organic compounds (VOCs) in air collected in specially prepared SUMMA canisters and analyzed by gas chromatography/mass spectrometry (GC/MS). <http://www.caslab.com/EPA-Methods/PDF/to-15r.pdf>

**TABLE 1**  
**Summary of Samples Submitted to EPA ORD**  
**for Poly- and Perfluoroalkyl Substances (PFAS) Analyses**  
**WORK PLAN #1**

Sample Material	Sample Number	Sample Collection Date	Sample Location Description	EPA ORD Report(s)	Type of Analyses Reported
Groundwater	199712055MWGZ1	5/31/2018	Monitoring Well	NR	Pending
	199712055MWGZ1 (duplicate)	5/31/2018	Monitoring Well	NR	Pending
	199712055MWGZ2	5/31/2018	Monitoring Well	NR	Pending
	199712055MWGZ3	5/31/2018	Monitoring Well	NR	Pending
	EPAORD_007	8/30/2017	Private Well	#3	Targeted PFAS; non-Targeted PFAS results pending
	EPAORD_008	8/25/2017	Private Well	#3	Targeted PFAS; non-Targeted PFAS results pending
	EPAORD_009	8/24/2017	Private Well	#3	Targeted PFAS; non-Targeted PFAS results pending
	EPAORD_010	8/31/2017	Private Well	#3	Targeted PFAS; non-Targeted PFAS results pending
	EPAORD_011	8/24/2017	Private Well	#3	Targeted PFAS; non-Targeted PFAS results pending
	EPAORD_012	8/25/2017	Private Well	#3	Targeted PFAS; non-Targeted PFAS results pending
	EPAORD_013 (dup)	8/25/2017	Private Well	#3	Targeted PFAS; non-Targeted PFAS results pending
	EPAORD_014	8/31/2017	Private Well	#3	Targeted PFAS; non-Targeted PFAS results pending
	EPAORD_015	8/29/2017	Private Well	#3	Targeted PFAS; non-Targeted PFAS results pending
	EPAORD_016	8/31/2017	Private Well	#3	Targeted PFAS; non-Targeted PFAS results pending
	EPAORD_017	8/31/2017	Private Well	#3	Targeted PFAS; non-Targeted PFAS results pending
	EPAORD_018	8/25/2017	Private Well	#3	Targeted PFAS; non-Targeted PFAS results pending
	EPAORD_019	8/30/2017	Private Well	#3	Targeted PFAS; non-Targeted PFAS results pending
	EPAORD_020	8/30/2017	Private Well	#3	Targeted PFAS; non-Targeted PFAS results pending
	EPAORD_021 (dup)	8/30/2017	Private Well	#3	Targeted PFAS; non-Targeted PFAS results pending
	EPAORD_022	9/8/2017	Private Well	#3	Targeted PFAS; non-Targeted PFAS results pending
Surface Water	EPAORD_023	9/8/2017	Private Well	#3	Targeted PFAS; non-Targeted PFAS results pending
	EPAORD_024	9/5/2017	Private Well	#3	Targeted PFAS; non-Targeted PFAS results pending
	EPAORD_901	9/8/2017	Private Well	#3	Targeted PFAS; non-Targeted PFAS results pending
	EPAORD_001	8/28/2017	Dumpling Brook, Upstream	#3	Targeted PFAS
	EPAORD_002	8/28/2017	Merrimack River, Downstream	#3	Targeted PFAS
	EPAORD_003	8/28/2017	Merrimack River, Upstream	#3	Targeted PFAS
Soil	EPAORD_004	8/28/2017	Dumpling Brook, Downstream	#3	Targeted PFAS
	EPAORD_005 (duplicate)	8/28/2017	Dumpling Brook, Downstream	#3	Targeted PFAS
	EPAORD_006	8/28/2017	Stormwater Outfall	#3	Targeted PFAS
Soil	EPAORDS1	9/1/2017	Soil Boring (0")	#1 & #2	Targeted PFAS; Non-Targeted PFAS
	EPAORDS2	9/1/2017	Soil Boring (2-4')	#1 & #2	Targeted PFAS; Non-Targeted PFAS
	EPAORDS3	9/1/2017	Soil Boring (4-6')	#1 & #2	Targeted PFAS; Non-Targeted PFAS
Char Material from Tower Stacks	MA Tower	August, 2017	Interior of stack	#1 & #2	Targeted PFAS; Non-Targeted PFAS
	MS Tower	August, 2017	Interior of stack	#1 & #2	Targeted PFAS; Non-Targeted PFAS
	QX Tower	August, 2017	Interior of stack	#1 & #2	Targeted PFAS; Non-Targeted PFAS
Raw Materials Used in Dispersions	1	August, 2017	Directly from container shipped by supplier	NR	Pending
	2	August, 2017	Directly from container shipped by supplier	NR	Pending
	3	August, 2017	Directly from container shipped by supplier	NR	Pending
	4	August, 2017	Directly from container shipped by supplier	NR	Pending
	5	August, 2017	Directly from container shipped by supplier	NR	Pending
	6	August, 2017	Directly from container shipped by supplier	NR	Pending
	7	August, 2017	Directly from container shipped by supplier	NR	Pending
	8	August, 2017	Directly from container shipped by supplier	NR	Pending
	9	August, 2017	Directly from container shipped by supplier	NR	Pending
	10	August, 2017	Directly from container shipped by supplier	NR	Pending
	11	August, 2017	Directly from container shipped by supplier	NR	Pending
	12	August, 2017	Directly from container shipped by supplier	NR	Pending
	13	August, 2017	Directly from container shipped by supplier	NR	Pending

**Acronyms**

PFOA	Perfluorocarboxylates are reported as perfluorocarboxylic acids.
PFAS	Per- and Polyfluoroalkyl substances
NR	Not Reported

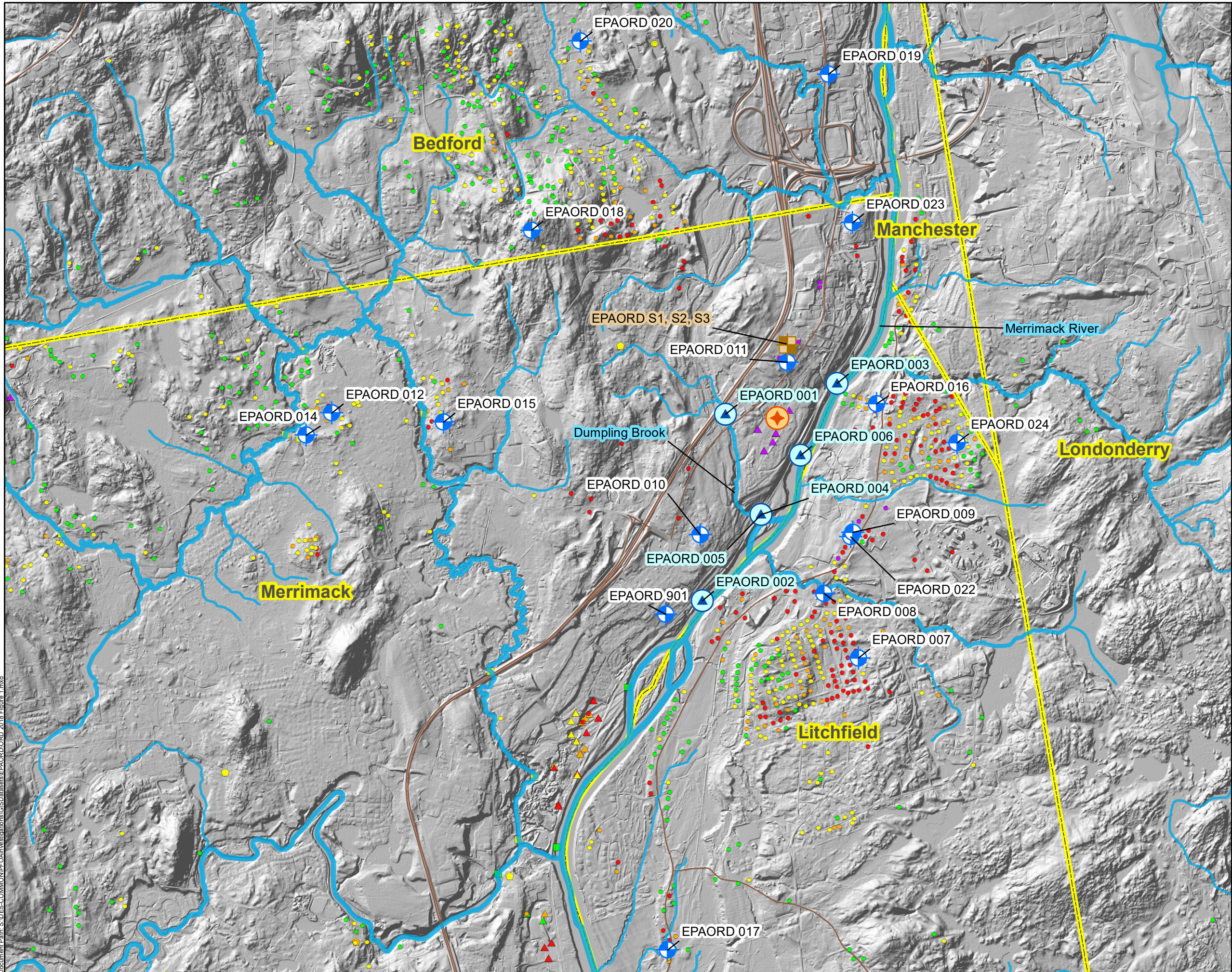
**TABLE 2**  
**Summary of Samples Submitted to EPA ORD**  
**for Poly- and Perfluoroalkyl Substances (PFAS) Analyses**  
**WORK PLAN #2**

Sample Material	Sample Number	Sample Collection Date	Sample Location Description	EPA ORD Report(s)	Type of Analyses Reported
Air Emission Samples	500-520	April 26 & 27, 2018	MA Tower	NR	Pending
	600-620	April 26 & 27, 2018	MS Tower	NR	Pending
	700-720	April 30 & May 1, 2018	QX Tower Inlet to Pilot-scale Control Device	NR	Pending
	800-820	April 30 & May 1, 2018	QX Tower Outlet of Pilot-scale Control Device	NR	Pending
SUMMA Canister Samples	Background 1		System Blank	#4	Non-Targeted PFAS; Volatile Organic HAPs
	RK9	5/1/2018	Ambient (inside facility)	#4	Non-Targeted PFAS; Volatile Organic HAPs
	005	5/1/2018	Ambient (lower roof)	#4	Non-Targeted PFAS; Volatile Organic HAPs
	794	4/27/2018	Ambient (upper roof)	#4	Non-Targeted PFAS; Volatile Organic HAPs
	709	4/30/2018	Ambient (Field Blank)	#4	Non-Targeted PFAS; Volatile Organic HAPs
	755	4/26/2018	MA Tower	#4	Non-Targeted PFAS; Volatile Organic HAPs
	751	4/27/2018	MA Tower	#4	Non-Targeted PFAS; Volatile Organic HAPs
	262	4/27/2018	MA Tower	#4	Non-Targeted PFAS; Volatile Organic HAPs
	068	4/26/2018	MS Tower	#4	Non-Targeted PFAS; Volatile Organic HAPs
	700	4/27/2018	MS Tower	#4	Non-Targeted PFAS; Volatile Organic HAPs
	744	4/27/2018	MS Tower	#4	Non-Targeted PFAS; Volatile Organic HAPs
	721	4/30/2018	QX Tower (inlet to Pilot-scale Control Device)	#4	Non-Targeted PFAS; Volatile Organic HAPs
	176	5/1/2018	QX Tower (inlet to Pilot-scale Control Device)	#4	Non-Targeted PFAS; Volatile Organic HAPs
	A378	5/1/2018	QX Tower (inlet to Pilot-scale Control Device)	#4	Non-Targeted PFAS; Volatile Organic HAPs
	321	4/30/2018	QX Tower (outlet to Pilot-scale Control Device)	#4	Non-Targeted PFAS; Volatile Organic HAPs
	2045	5/1/2018	QX Tower (outlet to Pilot-scale Control Device)	#4	Non-Targeted PFAS; Volatile Organic HAPs
	793	5/1/2018	QX Tower (outlet to Pilot-scale Control Device)	#4	Non-Targeted PFAS; Volatile Organic HAPs
	Background 2		System Blank	#4	Non-Targeted PFAS; Volatile Organic HAPs
Dip Tank Coatings	521	4/26/2018	MA Tower	NR	Pending
	522	4/27/2018	MA Tower	NR	Pending
	523	4/27/2018	MA Tower	NR	Pending
	621	4/26/2018	MS Tower	NR	Pending
	622	4/27/2018	MS Tower	NR	Pending
	623	4/27/2018	MS Tower	NR	Pending
	721	4/30/2018	QX Tower Dip Pan 1	NR	Pending
	722	4/30/2018	QX Tower Dip Pan 2-5	NR	Pending
	725	5/1/2018	QX Tower Dip Pan 1	NR	Pending
	726	5/1/2018	QX Tower Dip Pan 2-5	NR	Pending
Char Material from Tower Stacks	729	5/1/2018	QX Tower Dip Pan 1	NR	Pending
	MA Tower	5/2/2018	Interior of stack	NR	Pending
	MS Tower	5/2/2018	Interior of stack	NR	Pending
Process Water from Pilot-Scale APCE	QX Tower	5/2/2018	Interior of stack	NR	Pending
	733	5/1/2018	Supply Water	NR	Pending
	734	4/30/2018	Sump Water	NR	Pending
	735	5/1/2018	Sump Water	NR	Pending
	736	5/1/2018	Sump Water	NR	Pending

**Acronyms**

PFCA	Perfluorocarboxylates are reported as perfluorocarboxylic acids.
PFAS	Per- and Polyfluoroalkyl substances
NR	Not Reported
HAPs	Hazardous Air Pollutants





**Figure 1 - EPA ORD  
Sample Locations**

**EPA ORD Sample Locations**

- Char
- Soil
- Surface Water
- Groundwater

**PFOA + PFOS (PPT)**

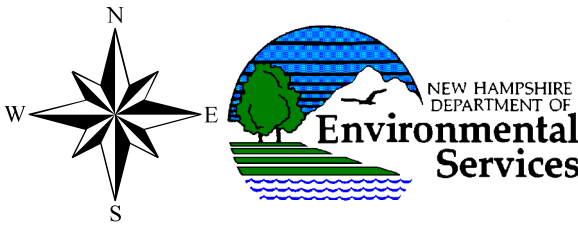
Supply Well	Monitoring Well	Surface Water	Other Sample	
				≥400
				70 - <400
				45 - <70
				10 - <45
				< 10

Town Boundary

0 0.25 0.5 1 Miles

1:30,000

1 inch = 2,500 feet



Map By: Jeffrey Marts and Samuel Fontaine



Appendix A-1

NHDES Letter – New Hampshire Department of Environmental Services  
Request for Assistance Assessing Poly- and Perfluoroalkyl Substances  
June 22, 2017





The State of New Hampshire  
**Department of Environmental Services**

**Clark B. Freise, Assistant Commissioner**



June 22, 2017

Jennifer Orme-Zavaleta, PhD  
Director, National Exposure Research Laboratory  
USEPA Office of Research and Development  
109 TW Alexander Dr MC 305-01  
RTP, NC 27711

**Subject: New Hampshire Department of Environmental Services Request for Assistance Assessing Poly- and Perfluoroalkyl Substances**

Dear Dr. Orme-Zavaleta:

The New Hampshire Department of Environmental Services (NHDES) is requesting assistance for completing analyses of long and short-chain poly and perfluoroalkyl substances (PFAS) in industrial chemicals, groundwater, surface water, soil, sludge, air, process residuals and potentially food crops surrounding two sites where air emissions associated with processes that historically used PFAS-containing raw materials. These sites have historically released PFAS into the environment and have contaminated soil and water, including groundwater used as drinking water for tens of thousands of people in the state. NHDES is also concerned that ongoing air emissions of certain PFAS may be occurring with minimal air pollution controls being applied.

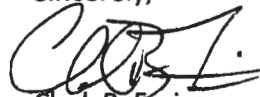
NHDES is currently using commercial laboratories to complete analyses that report results of 14 to 23 traditional PFAS compounds. NHDES has attempted to coordinate with a laboratory to analyze for a PFAS compound variant that was reported to have replaced PFOA at a facility that has ongoing air emissions containing PFAS. Despite analyzing over 1,000 samples from private and public drinking water wells in an area where groundwater has been contaminated with PFOA, this replacement compound has yet to be detected in water near this site. However, the replacement compound was detected in stack samples at the site.

Assistance from the United States Environmental Protection Agency (USEPA) is needed to test for environmental contaminants that are potentially being released to the air and impacting the environment. USEPA's assistance is needed to overcome technical barriers for completing this work to date including: 1) commercial laboratory analytical limitations for handling more complex sample matrices; 2) unknown nature of the compounds because they are proprietary, manufacturing byproducts or degradation compounds contained in raw materials; and 3) lack of expertise and experience associated with advanced fluorochemistry and fate and transport properties. USEPA could assist by analyzing samples using high resolution mass spectrometry with a comprehensive assessment of the spectral data using library searches.

NHDES will utilize this information to: 1) understand the multimedia environmental distribution of fluorinated compounds generated when emitting PFAS to the air; 2) ensure drinking water treatment systems and remediation systems being designed to remove PFOA and PFOS are able to also consider employing treatment that can remove other PFAS compounds and associated degradates, some which could be precursors to the formation of PFOA and PFOS. Granular activated carbon is the standard treatment technology for PFOA and PFOS, but is not as effective in removing some of the shorter chain PFAS compounds or precursors to PFOS and PFOA. This information is critical for ensuring drinking water is appropriately treated and that remediation systems removing PFOA and PFOS do not unknowingly redistribute other PFAS contaminants; 3) differentiate sources of contamination when and where there is the potential for multiple sources of contamination by identifying a signature of distribution of compounds for different sources of PFAS; 4) assess if conditions warrant the installation of treatment systems for air emissions to proactively prevent the contamination of the environment and drinking water with the newer alternative chemicals or precursor compounds; 5) prioritize what contaminant(s) need a risk assessment based on what is actually being measured in the environment, including drinking water.

We greatly appreciate your assistance on this matter. We look forward to our continued partnership in successfully addressing these emerging drinking water contaminants. Please do not hesitate to contact me ([Clark.Freise@des.nh.gov](mailto:Clark.Freise@des.nh.gov), (603)271-8806) or Brandon Kernan ([Brandon.Kernen@des.nh.gov](mailto:Brandon.Kernen@des.nh.gov), (603)271-0660) should you have any questions.

Sincerely,



Clark B. Freise

Assistant Commissioner

cc: Meghan Cassidy, USEPA Region 1  
Andy Lindstrom, USEPA ORD  
Michael Wimsatt, NHDES  
Eugene Forbes, NHDES  
Brandon Kernan, NHDES

## Appendix A-2

Work Plan #1 – Southern New Hampshire Sample Collection Plan for  
Non-Targeted Per- and Poly-fluorinated Compounds Analyses  
August 30, 2017

**Southern New Hampshire Sample Collection Plan for Non-Targeted Poly- and  
Perfluoroalkyl Substances (PFAS) Analyses**  
**August 30, 2017**

**1.0 Objective**

The objective of this sampling program is to identify the occurrence of the full spectrum of poly- and perfluoroalkyl substances (PFAS) in process feedstocks, product residuals, air emissions, surface water, groundwater and soil near two textile manufacturing facilities in southern New Hampshire. The identification of PFAS compounds will be completed by using a high resolution mass spectrometer at the United States Environmental Protection Agency's Office of Research and Development's lab at Research Triangle Park in Durham, NC. The information is needed to identify the specific PFAS compounds and their byproducts associated with air emissions that are being detected in the environment. NHDES will use this information to identify other target PFAS compounds in an effort to expand commercial labs analyte lists.

**2.0 Approach to Work**

The approach to completing the work is described in Tasks 1-8, below. The sampling locations associated with each task are shown in figure attached as a file named "Figure."

**Task 1 - Sample PFAS-Based Dispersion Products at Textile Coating Facilities**

Samples of raw dispersion products consisting of PFAS compounds will be collected at two textile coating facilities. Products will be sampled at the Saint-Gobain Performance Plastics (SGPPL) facility in Merrimack, NH and the Textile Coating International (TCI) facility in Manchester, NH. These thirteen dispersions represent all the raw dispersions currently in use at the two facilities.

**Task 2 – Sample Char/Carbon Material Taken from Air Emission Towers at SGPPL**

Three samples of solid materials that accumulate on the interior of the towers will be collected and are summarized as follows:

- One sample from the MA Tower which has been in operation at SGPPL since 1994. The ductwork was replaced and the oven and ancillary process components were cleaned in 2016. Therefore, the solid material that will be collected from this stack will most likely represent new dispersions used since 2016.
- One sample from the MS Tower which has been in operation at SGPPL since 2002. The solid material that will be collected from this stack may potentially contain residue components from pre-2006 PFOA based dispersions AND new dispersions that have been used since 2006.
- One sample from the QX Tower which has been in operation at SGPPL since 1989. This tower has the highest PFOA partition factor based on previous stack test results and therefore potentially

receives the highest load of emissions. The solid material that will be collected from this stack may potentially contain residue components from pre-2006 PFOA based dispersions AND new dispersions that have been used since 2006.

Summaries of analytical data associated with the dispersion materials and char material are included in the file named "Task 2 Attachments."<sup>1</sup>

### **Task 3 – Sample Highly Contaminated Groundwater and Soil Immediately Downgradient of the SGPPL Facility**

A water sample will be collected from a shallow groundwater monitoring well immediately adjacent to and downgradient of the SGPPL property. A soil boring will be advanced 6-8 feet deep and approximately four soil profile samples will be collected.

Summaries of analytical data associated with groundwater and soil sampling at the SGPPL facility are included in the file named "Task 3 Attachments."

### **Task 4 – Surface Water Sampling**

One water sample will be collected from the stormwater outfall that discharges stormwater from the SGPPL facility to the Merrimack River. Two samples of surface water will be collected from the Merrimack River up gradient and downgradient of the facility. Two water samples will be collected from Dumping Brook which flows into the Merrimack River near the SGPPL property.

A summary of analytical data associated with the storm water outfall associated with the SGPPL facility is included in the file named "Task 4 Attachments." Water quality data for the Merrimack River and Dumping Brook have not been collected to date.

### **Task 5 – Groundwater Sampling – Wells with Groundwater Exceeding 200 Parts-Per-Trillion PFOA**

Groundwater samples will be collected from four private wells near SGPPL that exceed 200 Parts-Per Trillion (ppt) for PFOA.

A summary of analytical data for all of the potential private wells that meet this criterion is included in the file named "Task 5 Attachments."

### **Task 6 - Groundwater Sampling – Wells with PFOA Concentrations between 50 and 100 ppt PFOA**

Groundwater samples will be collected from four private wells located within three miles of the SGPPL facility that exhibit PFOA concentrations between 50-100 ppt.

A summary of analytical data for all of the potential private wells that meet this criterion is included in the file named "Task 6 Attachments."

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<sup>1</sup> The sample numbering in the Task 2 Attachments do not necessarily correspond to the sample numbering used in this exercise.

### **Task 7 – Groundwater Sampling – Wells With Elevated PFOA and PFOS Concentrations and Located Near Additional Sources of PFAS Contamination**

Groundwater samples will be collected from six private wells that exhibit a combined concentration of PFOA and PFOS above 70 ppt and are located in areas that are: 1) Likely impacted by PFAS releases to air associated with SGPPL; and 2) Alleged to be potentially impacted by additional potential sources of PFAS.

A summary of analytical data for all of the potential private wells that meet this criterion is included in the file named “Task 7 Attachments.”

### **Task 8 – Groundwater Sampling – Merrimack Village District Wells 4 and 5**

A groundwater sample will be collected from both Merrimack Village District (MVD) Well 4 and MVD Well 5.

A summary of analytical data for MVD 4 and MVD 5 is included in the file named “Task 8 Attachments.”

The following types and quantities of samples will be collected for each project task.

	Groundwater	Surface Water	Soil	Char Material from Tower Stacks	Raw Dispersions
<b>Task 1</b>					13
<b>Task 2</b>				3	
<b>Task 3</b>	1		4		
<b>Task 4</b>		5			
<b>Task 5</b>	4				
<b>Task 6</b>	4				
<b>Task 7</b>	6				
<b>Task 8</b>	2				
<b>Task 9</b>					
<b>Total</b>	<b>17</b>	<b>5</b>	<b>4</b>	<b>3</b>	<b>13</b>
<b>Total Number of Samples&gt;&gt;&gt;</b>					<b>42</b>

### **3.0 Sampling Procedures and Quality Assurance Project Plan**

NHDES’ Quality Assurance Project Plan (QAPP) is included as an attached file. The procedures for sampling for PFAS in the QAPP begin on document page 246. Additional information describing the soil sample collection methodology is included in an attached file.

### **4.0 Schedule**

The sample collection for Tasks 1-8 described in this work plan shall be completed from August 28, 2017 – September 22, 2017. Sampling for tasks 1-8 will occur concurrently.

Appendix A-3

NHDES Letter – New Hampshire Department of Environmental Services  
Request for Additional Assistance Assessing Poly- and Perfluoroalkyl  
Substances

October 27, 2017





The State of New Hampshire  
**Department of Environmental Services**

Clark B. Freise, Assistant Commissioner



October 27, 2017

Timothy H. Watkins  
Acting Director, National Exposure Research Laboratory  
USEPA Office of Research and Development  
109 TW Alexander Dr MC 305-01  
RTP, NC 27711

**Subject: New Hampshire Department of Environmental Services Request for Additional Assistance  
Assessing Poly- and Perfluoroalkyl Substances**

Dear Mr. Watkins:

The New Hampshire Department of Environmental Services (NHDES) is requesting additional assistance for completing analyses of long and short-chain poly and perfluoroalkyl substances (PFAS) in industrial coating formulations and air emissions taken directly from the stacks located at a site where processes are currently utilizing PFAS-containing raw materials. This site has historically released PFAS into the environment and has contaminated soil and water, including groundwater used as drinking water for tens of thousands of people in the state. NHDES is concerned that ongoing air emissions of certain PFAS may be occurring in the absence of air pollution controls.

NHDES is currently using commercial laboratories to complete analyses that report results of 14 to 23 traditional PFAS compounds. NHDES has attempted to coordinate with a laboratory to analyze for a shorter chain PFAS compound variant that was reported to have replaced PFOA at the above reference facility. Despite analyzing over 1,000 samples from private and public drinking water wells in an area where groundwater has been contaminated with PFOA, this replacement compound has yet to be detected in water near this site. However, the replacement compound was detected in previous stack samples at the site and has been detected associated with other sites elsewhere in the country.

Assistance from the United States Environmental Protection Agency (USEPA) is needed to test for environmental contaminants that are potentially being released to the air and impacting the environment. USEPA's assistance is needed to overcome technical barriers for completing this work to date including: 1) commercial laboratory analytical limitations for handling more complex sample matrices; 2) unknown nature of the compounds because they are proprietary, manufacturing byproducts or degradation compounds contained in raw materials; and 3) lack of expertise and experience associated with advanced fluorochemistry and its fate and transport properties. USEPA could assist by analyzing samples using high resolution mass spectrometry with a comprehensive assessment of the spectral data using library searches.

[www.des.nh.gov](http://www.des.nh.gov)

29 Hazen Drive • PO Box 95 • Concord, NH 03302-0095  
(603) 271-3503 • Fax: 271-2867 • TDD Access: Relay NH 1-800-735-2964



NHDES will utilize this information to:

- (1) Understand the current emissions of fluorinated compounds generated when utilizing current chemical formulations;
- (2) Assess if conditions warrant the installation of treatment systems for air emissions to proactively prevent the contamination of the environment and drinking water with the newer alternative chemicals or precursor compounds;
- (3) Ensure any potential air pollution control equipment systems that may be required to be installed are designed to remove not only PFOA and PFOS compounds but also any other PFAS compounds and associated degradates, some which could be precursors to the formation of PFOA and PFOS. This information is critical for ensuring any air pollution control equipment is properly designed for potential PFAS emissions and that remediation systems removing PFOA and PFOS do not unknowingly redistribute other PFAS contaminants;
- (4) Differentiate sources of contamination when and where there is the potential for multiple sources of contamination by identifying a signature of distribution of compounds for different sources of PFAS; and
- (5) Prioritize what contaminant(s) need a risk assessment based on what is actually being measured in the environment via the air emission pathway.

We greatly appreciate your assistance on this matter. We look forward to our continued partnership in successfully addressing these emerging drinking water contaminants. Please do not hesitate to contact me ([Clark.Freise@des.nh.gov](mailto:Clark.Freise@des.nh.gov), (603)271-8806) or Catherine Beahm ([Catherine.Beahm@des.nh.gov](mailto:Catherine.Beahm@des.nh.gov), (603) 271-2822) should you have any questions.

Sincerely,



Clark B. Freise

Assistant Commissioner

cc: Meghan Cassidy, USEPA Region 1  
Andy Lindstrom, USEPA ORD  
Michael Wimsatt, NHDES  
Lea Anne Atwell, NHDES  
Eugene Forbes, NHDES  
Brandon Kernen, NHDES  
Michael Fitzgerald, NHDES  
Cathy Beahm, NHDES

## Appendix A-4

Work Plan #2 – Saint-Gobain Performance Plastics Air Sample Collection  
Plan for Non-Targeted Per- and Polyfluoroalkyl Substances Analysis  
April 18, 2018

**Saint-Gobain Performance Plastics Air Sample Collection Plan for Non-Targeted Per-  
and Polyfluoroalkyl Substances Analyses**  
**April 18, 2018**

**1.0 Objective**

The objectives of this sampling program are to identify the full spectrum of per- and polyfluoroalkyl substances (PFAS) in air emissions, process feedstocks, and process residuals from Saint-Gobain Performance Plastics (SGPPL) in Merrimack, New Hampshire and to assess the performance of a candidate pilot-scale air pollution control system. The identification and possible quantification of PFAS compounds will be conducted using multiple GC/LC/mass spectrometric techniques (e.g. low and high resolution time of flight preceded by gas chromatography separation and tandem mass spec preceded by liquid chromatography separation). These analyses will be conducted at the United States Environmental Protection Agency's Office of Research and Development (ORD) laboratories located at Research Triangle Park, North Carolina and Athens, Georgia.

The results of the program will be used to identify the specific PFAS compounds and related byproducts associated with air emissions and compounds that are being detected in the environment. NHDES will utilize this information to:

- (1) Document emissions of fluorinated compounds utilizing current chemical formulations;
- (2) Determine whether conditions warrant the installation of air pollution controls to prevent the environmental impact with the next generation of raw materials;
- (3) Ensure that a pilot scale air pollution control system is designed to effectively control PFAS and associated analogues, some of which are known precursors to the formation of PFOA and PFOS.
- (4) Develop "source type signatures" to differentiate multiple sources of contamination; and
- (5) Develop risk based prioritization procedures for evaluating air emissions (both deposition and inhalation pathways) for contaminant(s) measured in the environment.

**2.0 Approach to Work**

The approach to completing the work is described in Tasks 1-4, below. The sampling locations associated with each task are shown in the Figures 3 – 5 of the attached SGPPL Stack Test Plan dated April 11, 2018.

## Task 1 – Air Emission Samples

SGPPL has contracted with Barr Engineering to conduct the stack testing that will collect the air emission samples. Barr Engineering will be using SGS Laboratories to prepare field reagents and perform analytical work for SGPPL. NHDES staff will be on site during the stack testing to observe the entire test program and SGPPL has agreed to collect additional samples<sup>1</sup> for submittal to ORD for non-routine analyses.

The combined program will involve the testing of one cast film and two fabric coating towers as listed below:

- MA Tower – Emission samples from this fabric coating tower will be collected at the uncontrolled exhaust prior to the dilution fan/exhaust stack.
- MS Tower – Emission samples from this fabric coating tower will be collected at the uncontrolled exhaust prior to the dilution fan/exhaust stack.
- QX Tower – Emission samples from this cast film tower will be equipped with a pilot-scale wet cyclone/fiberbed mist collection system (APCE). Emission samples will be collected simultaneously at the inlet and outlet locations of the APCE.

A modified method 5 (MM5) train will be used to collect PFAS compounds with nominal boiling points greater than 100°C. For the purpose of collecting samples for ORD, three 2-hour test runs<sup>2</sup> will be conducted for each tower location for a total of 12 sample sets. In addition, reagent blanks will be collected for each MM5 fraction and a field biased blank sampling train will be set up and recovered to assess any field contamination issues.

Each MM5 sample train consists of a nozzle, heated probe, heated glass fiber filter, XAD-2/Condenser Module, three Greenberg-Smith impingers [one containing 100 mL DI water, one containing 100 mL 0.1N sodium hydroxide (NaOH), and one containing 0.01N sodium borate], a second unheated filter followed by an indicating silica gel impinger for water vapor removal. Since each train has 6 fractions plus a methanol (MeOH) rinse fraction, there will be a total of 7 fractions per sampling train for a total of 98 samples to be analyzed separately.

Summa Canisters will be used to collect volatile compounds with boiling points less than 100°C. Canister samples will be collected during each of the 2-hour test runs summarized above. Sampling will be conducted in accordance with the methodology specified in EPA's *Compendium of Methods for the Determination of Toxic Organic Compound – TO14A and TO15*. Canister orifices will be adjusted to

---

<sup>1</sup> Since the tower ductwork is anticipated to be less than 24 inches in diameter, concurrent sampling following standard EPA methodology is prohibitive. Therefore, a total of 6 test runs will be conducted for each tower sample location with every other test run sent to ORD and the remaining sent to the commercial lab (SGS Laboratory).

<sup>2</sup> Test runs will be two hours in duration unless labs state that longer timeframes are required for lower detection limits.

collect integrated samples during the specified 2-hour test period. NHDES will conduct the sampling according to EPA's Miscellaneous Operating Procedure (MOP) that will be provided as part of the Summa Canister shipment. A total of 12 samples, 1 field blank and 3 ambient locations (TBD) will be collected.

The industry standard when conducting stack testing using sampling trains involving resins is for the test company to send the glass resin traps and glass fiber filters to the lab conducting the sample analyses. The lab cleans the glass traps according to their QC protocol, prepares and QC's the resin batch (in this case XAD-2), packs the resin traps, spikes the traps with surrogates to assess recovery and ships the sampling media to the field for the sampling team to use. Similarly, the laboratory may QC the filter media/reagents and/or pre-clean the filters and provide these reagents to the sampling team. This is important because the sampling company must choose glass traps that are compatible with their sampling equipment. Also, on past tests for PFAS, the lab provided the other reagents that were QC'd as is the case with the XAD. This included the DI water, 0.1N NaOH, MeOH, 0.01N sodium borate, pre-cleaned glass fiber filters and sample recovery bottles.

In this case, there are two labs conducting the post-testing analyses: ORD and SGS Laboratories. Therefore, it is imperative that coordination of the pre-test preparation work be facilitated in a way that all parties are aware of the details and schedule. Barr Engineering will provide XAD glassware to SGS Laboratories for cleaning, packing resin, and spiking and SGS will provide the reagents for samples that are QC'd. The field team will collect reagent blanks in the field and assemble and recover a field-biased blank train to account for any handling bias during one of the test runs.

ORD will provide a surrogate mixture to SGS and SGS will add this surrogate mixture as a field spike to the resin traps designated for the ORD sample sets. These traps will be spiked with the labeled compounds listed below:

<b><u>Compound</u></b>	<b><u>Abbreviation</u></b>
Perfluoro-n-[3,4,5- <sup>13</sup> C <sub>3</sub> ]pentanoic acid	M3PFPeA
Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]decanoic acid	MPFDA
Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanoic acid	MPFOA
Sodium perfluoro-1-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanesulfonate	MPFOS
2-Perfluorohexyl-[1,2- <sup>13</sup> C <sub>2</sub> ]-ethanol (6:2)	M2FHET
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)- <sup>13</sup> C <sub>3</sub> -propanoic acid	M3HFPO-DA

## **Task 2 – Sample PFAS-Based Raw Materials**

Samples of raw materials thought to contain PFAS compounds were collected at this facility as part of the initial ORD sampling project in the fall of 2017. Based on process information submitted by SGPPL to NHDES, the raw materials that are planned to be used at the time of the stack testing are the same as those already sent to ORD. The actual coating formulations used at the time of the stack test will be collected from each dip pan(s) on each tower during each test run and sent to ORD for analysis. At a minimum, 1 sample from the dip pan from the MA and MS Towers for each of the stack test runs will be collected, for a total of 6 samples from these two towers. At a minimum, 1 sample from each dip pan from the QX Tower for each of the stack test runs will be collected. Since the QX Tower will be operated using four passes, there will be 4 samples for each stack test of the QX Tower for a total of 12 samples. In total, ORD will receive 18 dip pan samples.

## **Task 3 – Sample Char/Carbon Material Taken from Air Emission Towers**

Three samples of solid materials that accumulate on the interior of the towers were collected as part of the initial ORD sampling project in the fall of 2017. Three additional samples of this material will be collected during the stack testing program and are summarized as follows:

- One sample from the MA Tower which has been in operation at SGPPL since 1994. The ductwork was replaced and the oven and ancillary process components were cleaned in 2016. Therefore, the solid material that will be collected from this stack will most likely represent new dispersions used since 2016.
- One sample from the MS Tower which has been in operation at SGPPL since 2002. The solid material that will be collected from this stack may potentially contain residue components from pre-2006 PFOA based dispersions AND new dispersions that have been used since 2006.
- One sample from the QX Tower which has been in operation at SGPPL since 1989. This tower has the highest PFOA partition factor based on previous stack test results and therefore receives the highest load of emissions. The solid material coating the interior of the tower would likely be associated with emissions that occurred while PFOA-based dispersions were in use and emissions that occurred after the use of PFOA had been phased out.

## **Task 4 – Sampling of the Pilot-Scale Air Pollution Control Equipment (APCE)**

SGPPL is planning on conducting a pilot-scale evaluation of a candidate air pollution control technology on the QX Tower in addition to collecting samples for ORD. Inlet and outlet samples will be collected as described under Task 1 above.

The candidate APCE is a wet cyclone/fiberbed mist collection system. Gas enters the unit and passes through a wet cyclone where larger particles are removed. The material that is removed falls into the cyclone sump and the water in the sump is recirculated through the cyclone. In a full scale system, the sump has a makeup water feed and a slow solids removal cycle for higher solids applications. In the case of water soluble PFAS, the situation exists where the PFAS concentrations in the water could exceed the solubility of the sump liquid if the makeup water input is not sufficient.

The filter system is a spun fiber type (depth filter type) and would not normally have a pulse system to clean the surface of collected particulate. Any particulate that is not embedded in the filter structure drops to the hopper and the fine particles enter the depths of the filter. At some point, the filter plugs and must be changed.

In conjunction with the APCE evaluation and in addition to the air samples, NHDES will collect samples of the make-up water (from plant water source – 1 sample), sump water (for each of the three APCE test runs on QX Tower – 3 samples) and any solid matter that can be removed from the internals of the unit after the test is done and the unit is taken offline (e.g. sump solids, particulate filter material – 2 samples).

The following types and quantities of samples will be collected for each project task:

	<b>Air Emission Samples</b>	<b>Summa Canister Samples</b>	<b>Dip Tank Coatings</b>	<b>Char Material from Tower Stacks</b>	<b>Process Water and Solids from APCE</b>
<b>Task 1</b>	<b>98</b>	<b>16</b>			
<b>Task 2</b>			<b>18</b>		
<b>Task 3</b>				<b>3</b>	
<b>Task 4</b>					<b>6</b>
<b>Total</b>	<b>98</b>	<b>16</b>	<b>18</b>	<b>3</b>	<b>6</b>
	<b>Total Number of Analytical Fractions</b>				<b>141</b>

### **3.0 Sampling Procedures and Quality Assurance Project Plan**

NHDES' Quality Assurance Project Plan (QAPP) was included in the initial ORD request package. The procedures for sampling for PFAS in the QAPP begin on document page 246.

### **4.0 Schedule**

The sample collection described in this work plan shall be conducted late April and early May, 2018. Sampling for Tasks 1-4 will occur concurrently.

Appendix A-5  
Stack Test Plan Saint Gobain Performance Plastics Corporation  
Merrimack, New Hampshire  
April 11, 2018



## STACK TEST PLAN

### SAINT GOBAIN PERFORMANCE PLASTICS CORPORATION MERRIMACK, NEW HAMPSHIRE

Date test plan

created/revised/finalized: March 1, 2018 / April 2, 2018 / April 11, 2018

Scheduled test date(s): April 26-27 and April 30-May 2, 2018

#### PART I. GENERAL INFORMATION

Name and address of emission facility: Saint Gobain Performance Plastics Corporation  
701 Daniel Webster Highway  
Merrimack, NH 03054

Air Emission Facility ID Number: 3301100165

Facility Contact: Ed Canning (518) 345-2122  
Director, Health, Safety & Environment

Reason for the test: There are several objectives being undertaken in this stack test mobilization:

The first objectives are in response to the State of New Hampshire Department of Environmental Services (NHDES) request in a letter dated October 27, 2017 and subsequent meetings and conversations held since that time

1. Conduct Modified EPA Method 5 (MM5) stack emission testing on the MA and MS Coating Towers and the QX Cast Film exhausts.
2. Dip pan samples of formulated dispersions will be taken during each stack test run.
3. Determine UCMR 3 List 1 PFAS compounds and GenX stack emissions from the MA and MS Coating Towers and the QX Cast Film exhaust and dispersion samples with analytical services provided by SGS Accutest.
4. Measure NHDES regulated toxic air pollutants (RTAPs) ammonia and total fluorides as fluorine stack emissions from the MA Coating Tower exhaust. Samples to be submitted to Element One, Inc.

Additional objectives are included under the direction of Saint Gobain Performance Plastics

5. Conduct additional MM5 sample collection at the outlet of a coalescing fiberbed filtration control device to be installed on the QX Cast Film on a pilot basis for this test mobilization. This objective will be performed in

concert with objective 1 above, in that the inlet and outlet of the control device will be tested simultaneously and the inlet measurement will represent uncontrolled emissions.

6. A sample of the incoming water supply to the control device will be taken at the beginning of the test. Control device water sump samples will be taken during each stack test run. These samples will be submitted to SGS Accutest.
7. Determine UCMR 3 List 1 PFAS compounds and GenX stack emissions from the MA and MS Coating Towers and the QX Cast Film exhaust samples with analytical services provided by SGS Accutest.

All tests will be conducted under representative operating conditions with product and product coatings to be determined prior to the test mobilization. The selected products and coatings will be submitted to NHDES under separate, confidential cover as Confidential Business Information (CBI).

The MA and MS tower processes are fabric coating applications involving a preparation of aqueous fluoropolymer dispersion coating, coating application to a glass cloth web in a dip pan, and finally heat treating in three stages—dry zone, bake zone and fuse zone. The products being manufactured require multiple coating and heat treating applications. The tests are usually conducted during the first coating pass, as the first pass is the heaviest application of coating. The formulated coating, consisting of aqueous fluoropolymer dispersion surfactants and other processing aids, is sampled at the drip pan during the tests. Hot gases captured from the three stages of heating are exhausted through the tower stack.

QX cast film production involves a multi-coat process where formulated dispersion is coated in a dip pan on a web at room temperature, and then passed through a vertical oven or tower, similar to the glass cloth coating process, where the water and is removed. The web path through the tower is typically vertical. Multiple dipping and drying/baking steps may be used to produce a multi-layer film that is peeled from the web, resulting in the final product. The film is sintered like other fluoropolymer products to achieve final characteristics. Once the final film layer is applied, the film is wound onto a roller.

Diagrams of the glass cloth coating and the cast film processes are provided in Figures 1 and 2, respectively. Stack test sample port locations are provided in Figures 3-5.

Testing  
Company and  
Contact:

Barr Engineering Co. (952) 832-2630  
Tim Russell (612) 741-6889 mobile  
Vice President, Chemical Engineer  
trussell@barr.com  
4300 MarketPointe Drive, Suite 200  
Minneapolis, MN 55435

Private  
Analytical  
Laboratories and  
Contacts:

**PFAS and GenX analysis**  
SGS Accutest (407) 425-6700 ext 2602  
Norm Farmer (407) 595-9987 mobile  
Corporate Technical Director  
Norman.Farmer@SGS.com  
4405 Vineland Rd  
Suite C-15  
Orlando, FL 32811

**Ammonia and Total Fluorides analysis**  
Element One Inc.  
Paula Smith  
6319-D Carolina Beach Road  
Wilmington, NC 28412

## PART II. TESTING REQUIREMENTS

The table below provides a summary of the pollutants to be tested and test methods.

<b>Source Description</b>	<b>Limitation Basis of Pollutant Tested</b>	<b>Pollutant Tested and Applicable Emission Limit</b>	<b>Specific Methods Citation</b>
MA Coating Tower Exhaust	None	Determine UCMR 3 List 1 PFAS compounds and GenX. See compound list below.  Total Fluorides Ammonia	EPA Methods 1-4 Modified EPA Method 5 (Three 2-hour test runs)  EPA Method 13B (Three 1-hr test runs) EPA CTM 027 (Three 1-hr test runs)
MS Coating Tower Exhaust	None	Determine UCMR 3 List 1 PFAS compounds and GenX. See compound list below.	EPA Methods 1-4 Modified EPA Method 5 (Three 2-hour test runs)
QX Cast Film Line (pilot control device inlet and outlet)	None	Determine UCMR 3 List 1 PFAS compounds and GenX. See compound list below.	EPA Methods 1-4 Modified EPA Method 5 (Three 2-hour test runs) EPA Method 2 airflow rates will be measured on the main stack once during each test run to determine total airflow volume from the line. Moisture content, gas composition and pollutant concentration will be assumed equal to measurements made at the pilot scale test inlet location. The sum of airflow measurements made

			at the pilot scale test inlet and main stack will be used to calculate uncontrolled mass emissions from the line.
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**Fluorinated compound analyte list for SGS Accutest submitted samples:**

Perfluorooctane Sulfonic Acid (PFOS)

Perfluorooctanoic Acid (PFOA)

Perfluorononanoic Acid (PFNA)

Perfluorohexane Sulfonic Acid (PFHxS)

Perfluoroheptanoic Acid (PFHpA)

Perfluorobutane Sulfonic Acid (PFBS)

2,3,3,3-Tetrafluoro-2-(heptafluoropropoxy)propanoic acid (GenX)

**PFAS Detection Limits**

SGS Accutest provided information regarding current detection limits for the compounds targeted in this project. For most compounds, method detection limits (MDLs) are 0.0025 µg/l and reporting limits (RLs) are 0.010 µg/l. With that, a simple case of a 100 ml liquid sample fraction from an impinger sample with a typical air sample volume of 60 ft<sup>3</sup> in a two hour test run would have a detection limit of 0.59 ng/m<sup>3</sup> at the RL. Of course, the answer to the detection limit question becomes more complicated under the chosen sampling and analytical methodology. Each test run will generate seven sample fractions that are analyzed separately. For each test run, the reported total mass of a compound and resulting calculated air concentration and mass emission rate for that compound is the sum of the values determined for each sample fraction. The commonly accepted reporting convention is to add the values of all sample fractions with a detectable quantity of the compound plus the value at the detection limit for fractions that are non-detect and to qualify the calculated sum with a "<" designation.

### PART III. OPERATING CONDITIONS AND PROCESS SAMPLES

The table and paragraph below identifies the process equipment to be tested, the operating parameters to be monitored and reported, and the rationale for testing.

Process Equipment Description for Units to be Tested	MA and MS Coating Towers
Process Equipment Parameter Monitoring During Performance Test	Dispersion identification, dispersion application rate, web width, line speed, coating pass number and oven zone temperature information will be recorded during the test
Process Monitoring Frequency	Continuously or at least every 15 minutes during test run
Personal Assigned to Record data	Saint Gobain staff
Control Equipment Description	These sources are uncontrolled
Control Equipment Operating Parameter During Performance Test	Not applicable
Process Samples	Dispersion sample taken from dip pan
Sampling Frequency	1 sample per test run
Personnel Assigned to Collect Samples	Barr staff

Process Equipment Description for Units to be Tested	QX Cast Film Line
Process Equipment Parameter Monitoring During Performance Test	Dispersion identification, dispersion application rate, web width, line speed and oven temperature information will be recorded during the test
Process Monitoring Frequency	Continuously or at least every 15 minutes during test run
Personnel Assigned to Record data	Saint Gobain staff
Control Equipment Description	Pilot scale coalescing fiberbed filter
Control Equipment Operating Parameter During Performance Test	Quench water rate and pressure drop across pre-filter (if applicable) and fiberbed filter. Details to be adjusted based upon pilot unit specifics and instrumentation package
Monitoring Frequency	Start and every 15 minutes during test run
Personnel Assigned to Record data	Saint Gobain or Barr staff
Process Samples	Dispersion - QX process line utilizes multiple "dip pans". Each dip pan will be sampled individually during each test run. These samples will be collected and analyzed independently  Supply water Scrubber Sump water
Sampling Frequency	1 sample per test run
Personnel Assigned to Collect Samples	Saint Gobain or Barr staff

#### PART IV. TEST AND ANALYTICAL METHODS

The following is a description of the test methods, number of test runs, length of test runs, and sampling rate for each analyte.

- A. EPA Method 1 for the location of sampling ports and points. One determination per test location.
- B. EPA Method 2 for stack gas velocity and volumetric flow rate. One determination per Modified EPA Method 5 test run.
- C. Modified EPA Method 3/3A to determine stack gas molecular weight. One determination per Modified EPA Method 5 test run. Integrated gas samples of approximately 120 minutes will be collected in Tedlar Bags during each test run from the exhaust of the dry gas meter. The dry stack gas will be analyzed with a Servomex 1440 oxygen and carbon dioxide analyzer calibrated to

EPA Method 3A/7E specifications. Ambient air or EPA protocol gas will be used for the oxygen span gas upper value. CEMS grade nitrogen will be used to zero instrumentation. All other calibration gases used will be EPA Protocol certified gases. An example calibration gas certification is provided in the attachments.

- D. EPA Method 4 for determination of moisture content in stack gas. One determination per Modified EPA Method 5 test run.
- E. EPA Method 5 Modified to collect perfluorinated compounds. The sample train will consist of a sample nozzle and heated sample probe and three impingers in series followed by an unheated glass fiber filter. The three impingers will be charged in order with 100 ml each of de-ionized water, 0.1N sodium hydroxide, and 0.01N sodium borate buffer. Each of the five resulting sample fractions will be recovered and analyzed separately for the determination of total target analyte mass. The filter will be collected separately from a methanol rinse of all components of the sample train. The modification of Method 5 follows test protocols previously accepted by the USEPA. A copy of the method, as published in the February 2005 QAPP for the Dispersion Processor Mass Balance Study Project is attached. The modified method was written specifically for the collection of APFO, but has since been utilized for sampling the class of compounds included in this test. Sampling times will be three runs of 120 minute duration for each test. Additional modifications are being made to this method for this test and are described below in item F.
- F. Additional modifications are being made to Method 5 sample train for this test in order to capture semi-volatile PFAS compounds. A heated glass fiber filter will be added to the oven section of the sample train followed by a water chilled coiled condenser, water chilled XAD2 resin trap and a knockout impinger prior to the impinger array and final filter described above. The trap will be spiked by SGS Acutest at 100 ng for M3PFPeA, M2PFDA, M2PFOA and M4PFOS. The heated filter and resin trap will be collected and submitted for analysis as two additional sample fractions for a total of 7 samples for submittal (front filter, XAD2 trap, DI impinger, sodium hydroxide impinger, sodium borate impinger, back filter and all components of train methanol rinse). Water collected in the knockout impinger will be combined with the DI impinger sample. All components of the sample train will be rinsed with methanol. The silica gel impinger will be weighed pre and post test runs and will not be stored in a container. Reagent blanks will be collected multiple times during the sampling campaign. Train blanks will be collected for each test location and will consist of setting up and recovery of complete sample train. Moisture determinations from the sample trains will be determined gravimetrically. The following train components will be weighed pre and post test run: XAD2 trap, knockout impinger, DI impinger,

- 0.1 sodium hydroxide impinger, 0.01 sodium borate impinger, final filter assembly and the dry column. All samples, with exception of dispersion samples, will be stored in cooled condition.
- G. SGS Accutest of Orlando, FL will perform the laboratory analysis of the air impinger, dispersion coating and control device water samples using high pressure liquid chromatography and tandem mass spectrometry (LC/MS/MS) in accordance with EPA Method 537 modified to accommodate the air emission testing procedures and to enhance method performance as per SGS SOPS.
- H. EPA Method 13B Determination of total fluoride emissions. Gas stream will be sampled isokinetically to account for possible water droplets or mist. Sampling times will be three runs of 60 minute duration for each test. The sampling train will be arranged and operated as described in the method with filtration taking place after the third impinger. The reagent and recovery solvent will be environmental grade water. The filter media will be Whatman #1 as listed in the method (See Method 13A 7.1.1.1). Sample will be recovered into HDPE sample storage bottles. Reagent blanks will be collected and analyzed. Temperature control of samples is not required for this method. Description of the sample train, setup, operation, recovery and analysis are located in EPA Method 13B. A copy of the method is not provided with this test plan submittal.
- I. EPA Conditional Test Method (CTM) 027 Determination of ammonia emissions Modified. Gas stream will be sampled isokinetically to account for possible water droplets or mist. Sampling times will be three runs of 60-minute duration for the test. The sample train will be as operated and arranged as described in the method, except that filtration will take place in oven. Filtration in the oven was chosen as opposed to the in-stack approach in the described in the method due to concerns of potential area blockage in small diameter stack. The probe and filter temperatures will be operated at temperatures consistent with EPA Method 5. This is expected to have no impact on results since filterable portion is not analyzed. The sample train will consist of three impingers as described in the method. Impingers one, two and three will be loaded with 100 mls 0.1N sulfuric acid. The third impinger will be analyzed separately for determination of breakthrough. Reagent blanks will be collected and analyzed. Temperature control of samples is required for this method. The above exceptions, notwithstanding, the test description of the sample train, setup, operation, recovery and analysis are located in CTM Method 027. A copy of the method is not provided with this test plan submittal.

## PART V. CALIBRATION METHODS AND PRETEST PREPARATIONS

- A. The EPA Method 5 Equipment will be calibrated prior to the test in accordance with the requirements in 40 CFR Part 60, Appendix A, EPA Quality Assurance Handbook for Air



Pollutant Measurement Systems: Volume III Stationary Sources Specific Methods and Barr Standard Operating Procedures.

- B. Pretest preparations for Modified Method 5 for PFAS compounds includes cleaning and methanol rinse of all sample train glassware components and the preparation of sampling reagents. Special care is made to eliminate all components of the sample train that may contain fluoropolymer constituents in order to avoid trace contamination of the samples from those components. Examples include avoiding the use of Teflon™ compression fittings or ferrules, sealing tape or sample jar liners, and fluoropolymer encapsulated O-ring seals.

#### PART VI. QUALITY ASSURANCE AND LABORATORY STANDARD OPERATING PROCEDURES

- A. SGS Accutest Orlando facility has developed standard operating procedures for use of EPA Method 537 for the purposes of this project. The SOPs are considered to be proprietary business information by SGS and can be shared upon execution of a confidentiality agreement.
- B. A complete review of the analytical laboratory recoveries, duplicates, and raw data by a member of Barr's Data Quality Assurance staff will be performed and presented in the test report.

#### PART VII. SAFETY

Emergency procedures are outlined in Saint Gobain's "Contractor Control Program" which can be reviewed upon arrival at the facility. In the event of a medical, fire or other emergency response, dial 911 and then the company contact.

#### PART VIII. TEST SCHEDULE

Day 1 Monday 4/23	Day 2 Tuesday 4/24	Day 3 Wednesday 4/25	Day 4 Thursday 4/26	Day 5 Friday 4/27	Day 6 Saturday 4/28
Travel	Travel	Facility Safety Meeting Pretest Coordination Equipment Setup	Test Towers MA and MS  Three test runs on each tower	Test Towers MA and MS  Three test runs on each tower	Move gear from MA/MS to QX inlet/outlet

Day 7 Sunday 4/29	Day 8 Mon 4/30	Day 9 Tuesday 5/1	Day 10 Wednesday 5/2	Day 11 Thursday 5/3	Day 12 Friday 5/4
Day Off	Test QX Cast Film inlet/outlet  Three test runs simultaneous at each test location	Test QX Cast Film inlet/outlet  Three test runs simultaneous at each test location	Test Tower MA Fluorides and ammonia RTAP  Three 1-hr test runs per test (six runs total)	Travel	Travel

Typical Daily Test Schedule:

0700 Barr staff arrive and process is running at this time  
0800 Target test start time  
0800-1000 Test Run 1  
1000-1100 Sample recovery  
1100-1300 Test Run 2  
1300-1400 Sample recovery  
1400-1600 Test Run 3

## PART IX. TEST REPORT

One complete test report will be submitted within 45 days of receipt of the full and final laboratory data report.

Submittal Address:     Mr. Ed Peduto  
                                 Sr. Compliance Assessment Engineer  
                                 Air Resources Division – Compliance Bureau  
                                 New Hampshire Department of Environmental Services  
                                 29 Hazen Drive  
                                 Concord, NH 03301  
                                 (603) 271-1987  
                                 [Edward.PedutoJr@des.nh.gov](mailto:Edward.PedutoJr@des.nh.gov)

### Attachments:

Standard Operating Procedure- Particulate and Condensable Matter Sampling for Ammonium  
                                 Perfluoro-octanoate in Stack Emissions

Figure 1 – Typical Glass Cloth Process Diagram

Figure 2 – Typical Cast Film Process Diagram

Figure 3 – Test port locations Tower MA exhaust

Figure 4 - Test port locations Tower MS exhaust

Figure 5 – Test port locations QX cast film pilot control device inlet/outlet

Example Calibration Gas Certification

# **STANDARD OPERATING PROCEDURE**

## **Particulate and Condensable Matter Sampling for Ammonium Perfluoro-octanoate in Stack Emissions**

### **1.0 Scope and Application**

This Standard Operating Procedure (SOP) is applicable to gas streams flowing in ducts, stacks and flues for the determination of average flow velocity, moisture content, gas molecular weight (carbon dioxide (CO<sub>2</sub>), oxygen (O<sub>2</sub>) content) and ammonium perfluoro-octanoate (APFO) content in particulate and condensable matter in stationary sources.

This SOP is primarily based on USEPA Method 5, however it also incorporates procedures from USEPA Methods 1, 2, 3, 3A, and 4. It is the responsibility of the team leader/project manager to read and understand the EPA Methods referenced.

### **2.0 Summary of Method**

A representative measurement site is selected and stack gas is withdrawn isokinetically from the source and collected in a heated sample probe, a series of chilled glass impingers and on a glass fiber filter maintained at ambient temperature. The APFO mass is determined analytically; which includes any material that condenses at the operating temperatures of the sampling train, is scrubbed or filtered from the sample, or combines chemically with the sample train absorbing solutions.

### **3.0 Equipment and Supplies**

#### **3.1 Airflow Velocity**

Stainless steel standard and type S Pitot Tubes of various lengths

Calibrated Pyrometer

Water for Wet Bulb

Calibrated Thermocouples

Calibrated Barometer

Calibrated differential pressure measurement devices

Tape Measure

Duct tape

Sharpie Marking Pens

High temperature fiberglass tape

Wet Bulb Wicks

Spare batteries for pyrometer and electronic manometers

Protractor for Cyclonic Flow Determinations

### **3.2 APFO Sampling**

Stainless Steel Probes/Pitot tube assemblies (various lengths that include the probe sheath, S-type pitot tube, and stack temperature thermocouple)

Sample Modules (heated oven box and impinger ice bath) Umbilical (various lengths between 50 and 200 feet) Control Modules

Vacuum Pumps

Lab Box : Containing probe cleaning brush, field balance, sample recovery solvent wash bottles, desiccant, sample nozzle set with calipers for field validation of nozzle opening diameter and supply of compression fittings and ferrules for probe/nozzle union, 2- 10" crescent wrenches, 3- 250 ml polyethylene graduated cylinders, supply of deionized water (for purposes of this SOP when deionized water is referred to it means Type 1 water that has been treated with Hypercarb to remove traces of APFO), supply of acetone (for purposes of this SOP when acetone is referred to it means Optima Grade Acetone or equivalent), sample bottle labels, supply of pump and manometer oils, size 0 and 00 rubber stoppers, field portable pH meter and pH paper, duct tape, high temperature fiberglass tape, stopcock grease (acetone-insoluble, heat-stable silicone grease), neoprene gloves, leather and cloth work clothes, Kim wipes, spare fuse supply for control module, tape measure, 1/4" I.D latex tubing, spare silicone O-rings for ground glass ball fittings on glassware.

Glassware Box: containing glass filter holder assemblies, glass filter frits with silicone gaskets, filter cyclones and Erlenmeyer flasks, filter and cyclone bypass glassware, a supply of clean glass fiber filters in styrene Petri dishes, filter forceps, nylon bristle brushes and rubber policeman for sample recovery, size 0 and 00 rubber stoppers to seal glassware openings, Kim wipes.

Impinger sets (ball and socket type with silicone O-ring seals) with connecting u-tube glassware and glassware clamps.

Monorails and port clamps

Polyethylene

Sample containers

Tap Water

Toweling (cloth and fiberglass cloth) to seal sample ports during sample runs.

Probe Liners. Borosilicate or quartz glass tubing with a heating system capable of maintaining a probe gas temperature of  $(248 \pm 25^\circ\text{F})$  during sample performance.

Supply of 0.1N NaOH, 0.01N Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub> buffer solution, and methanol sampling reagents. Both the NaOH solution and the buffer solutions will be prepared in the laboratory using Type I Water that has been treated with Hypercarb to remove traces of APFO. For purposes of this SOP, when methanol is referred to it means Optima Grade Methanol or equivalent.

Crushed or cubed ice.

### **3.3 CO<sub>2</sub>, O<sub>2</sub> Determination**

Field portable oxygen meter to monitor oxygen content of the exhaust of the EPA Method 5 sample train during sample collection.

Leak-free bags (5-40 liters in size, Tedlar or similar construction) for the collection of integrated stack gas samples from the exhaust of the EPA Method 5 sample train during each sample run.

Orsat analyzer with reagent (oxygen absorbent and carbon dioxide absorbent) supply to support the performance of EPA Method 3 or oxygen/carbon dioxide gas analyzer(s) for EPA Method 3A. The choice of EPA Methods 3 or 3A is left to the discretion of the team leader.

Gas manifold for the introduction of calibration gas and the integrated gas sample for use with oxygen/carbon dioxide gas analyzer(s) in accordance with EPA Method 3A.

EPA Protocol 1 calibration gas mixtures for use with oxygen/carbon dioxide gas analyzer(s) in accordance with EPA Method 3A. Ambient air may be used as upper span gas for oxygen analyzer calibration. CEMS grade nitrogen will be used for both oxygen and carbon dioxide instrument zero.

### **3.4 Moisture Determination**

Same as APFO sampling equipment. The moisture determination is an intrinsic part of the APFO sample collection method and will be performed during each sample run.



### **3.5 General Equipment**

Extension cords (50 and 100 foot lengths) Lights

Shelter (tent) for outdoor sampling locations

Heater for cold weather sampling locations

Folding table and chair for equipment setup

Ropes (50-150 feet in length as needed) to raise and lower equipment to sample platforms

Plastic trash bags

Briefcase with copies of necessary field data sheets, clipboard, calculator, pens.

Laptop computer for field data entry using spreadsheet for EPA Method 5 calculations

Duct Tape

Tools

Two-way radios

Polyethylene Tubing (1/4" O.D.)

Supply of compression fittings

Sample Cooler Ice Cooler

### **3.6 Safety Equipment**

Hot gloves

Cold weather gear as needed

Hard Hats (with visors for splash protection as needed)

Protective coveralls and gloves as dictated by conditions at each sample location

Ear Plugs

Safety Footwear

Respiratory protection

Portable Eyewash

First Aid Kit

Safety Glasses

Harness and lanyard(s) as required to work safely at each sampling location

## **4.0 Procedure**

### **4.1 Selection of Measurement Site**

**4.1.1** Sampling and/or velocity measurements are performed at a site located at least eight stack or duct diameters downstream and two diameters upstream from any flow disturbance such as a bend, expansion,

or contraction in the stack, or from a visible flame. If necessary, an alternative location may be selected, at a position at least two stack or duct diameters downstream and a half diameter upstream from any flow disturbance. The procedure to select the measurement site is performed once for each sampling location.

#### **4.1.2 Particulate Traverses**

**4.1.2.1** When the eight- and two-diameter criterion can be met, the minimum number of traverse points shall be: (1) twelve, for circular or rectangular stacks with diameters (or equivalent diameters) greater than 0.61 meter (24 in.); (2) eight, for circular stacks with diameters between 0.30 and 0.61 meter (12 and 24 in.); and (3) nine, for rectangular stacks with equivalent diameters between 0.30 and 0.61 meter (12 and 24 in.).

**4.1.2.2** When the eight- and two-diameter criterion cannot be met, the minimum number of traverse points is determined from Figure 1-1 in EPA Method 1 (40 CFR Part 60 Appendix A). Before referring to the figure, however, determine the distances from the measurement site to the nearest upstream and downstream disturbances, and divide each distance by the stack diameter or equivalent diameter, to determine the distance in terms of the number of duct diameters. Then, determine from Figure 1-1 the minimum number of traverse points that corresponds: (1) to the number of duct diameters upstream; and (2) to the number of diameters downstream. Select the higher of the two minimum numbers of traverse points, or a greater value, so that for circular stacks the number is a multiple of 4, and for rectangular stacks, the number is one of those shown in Table 1-1 in EPA Method 1 (40 CFR Part 60 Appendix A).

#### **4.1.3 Cross-Sectional Layout and Location of Traverse Points.**

##### **4.1.3.1 Circular Stacks.**

**4.1.3.1.1** Locate the traverse points on two perpendicular diameters according to Table 1-2 in EPA Method 1 (40 CFR Part 60 Appendix A).

**4.1.3.1.2** For particulate traverses, one of the diameters must coincide with the plane containing the greatest expected concentration variation (e.g., after bends); one diameter shall be congruent to the direction of the bend.

**4.1.3.1.3** In addition, for elliptical stacks having unequal perpendicular diameters, separate traverse points shall be calculated and located along each diameter. To determine the cross-sectional area of the elliptical stack, use the following equation:

$$\text{Square Area} = D1 \times D2 \times 0.7854$$

Where:

D1 = Stack diameter 1

D2 = Stack diameter 2

**4.1.3.1.4** In addition, for stacks having diameters greater than 0.61 m (24 in.), no traverse points shall be within 2.5 centimeters (1.00 in.) of the stack walls; and for stack diameters equal to or less than 0.61 m (24 in.), no traverse points shall be located within 1.3 cm (0.50 in.) of the stack walls.

#### **4.1.4 Stacks with Diameters Greater Than 0.61 m (24 in.).**

**4.1.4.1** When any of the traverse points as located in Section 11.3.1 fall within 2.5 cm (1.0 in.) of the stack walls, relocate them away from the stack walls to: (1) a distance of 2.5 cm (1.0 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger. These relocated traverse points (on each end of a diameter) shall be the "adjusted" traverse points.

**4.1.4.2** Whenever two successive traverse points are combined to form a single adjusted traverse point, treat the adjusted point as two separate traverse points, both in the sampling and/or velocity measurement procedure, and in recording of the data.

#### **4.1.5 Stacks With Diameters Equal To or Less Than 0.61 m (24 in.).**

Follow the procedure in Section 4.1.3.1, noting only that any "adjusted" points should be relocated away from the stack walls to: (1) a distance of 1.3 cm (0.50 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger.

#### **4.1.6 Rectangular Stacks.**

**4.1.6.1** Determine the number of traverse points as explained in Sections 4.1.2.1 and 4.1.2.2 of this SOP. From Table 1-1, determine the grid configuration. Divide the stack cross-section into as many equal rectangular elemental areas as traverse points, and then locate a traverse point at the centroid of each equal area according to the example in Figure 1-4 in EPA Method 1 (40 CFR Part 60 Appendix A).

#### **4.1.7 Verification of Absence of Cyclonic Flow.**

**4.1.7.1** In most stationary sources, the direction of stack gas flow is essentially parallel to the stack walls. However, cyclonic flow may exist (1) after such devices as cyclones and inertial demisters

following venturi scrubbers, or (2) in stacks having tangential inlets or other duct configurations which tend to induce swirling; in these instances, the presence or absence of cyclonic flow at the sampling location must be determined. The following techniques are acceptable for this determination.

**4.1.7.2** Level and zero the manometer. Connect a Type S pitot tube to the manometer and leak-check system. Position the Type S pitot tube at each traverse point, in succession, so that the planes of the face openings of the pitot tube are perpendicular to the stack cross-sectional plane; when the Type S pitot tube is in this position, it is at "0° reference." Note the differential pressure (dp) reading at each traverse point. If a null (zero) pitot reading is obtained at 0° reference at a given traverse point, an acceptable flow condition exists at that point. If the pitot reading is not zero at 0° reference, rotate the pitot tube (up to ±90° yaw angle), until a null reading is obtained. Carefully determine and record the value of the rotation angle to the nearest degree. After the null technique has been applied at each traverse point, calculate the average of the absolute values of the rotation angle; assign values of 0° to those points for which no rotation was required, and include these in the overall average. If the average value of is greater than 20°, the overall flow condition in the stack is unacceptable, and alternative methodology, subject to the approval of the Administrator, must be used to perform accurate sample and velocity traverses.

#### **4.1.8 Data Analysis and Calculations.**

Nomenclature.

L = length  
W = width.

**4.1.8.1** For a rectangular cross section, an equivalent diameter (De) shall be calculated using the following equation, to determine the upstream and downstream distances:

$$De = 2 (L) (W)/(L + W)$$

## **4.2 Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)**

**4.2.1** Set up the apparatus as shown in Figure 2-1 in EPA Method 2 (40 CFR Part 60 Appendix A). Perform a leak-check be conducted as follows: (1) blow through the pitot impact opening until at least 7.6 cm (3.0 in.) H2O velocity head registers on the manometer; then, close off the impact opening. The pressure shall remain stable for at least 15 seconds; (2) do the same for the static pressure side, except using suction to obtain the minimum of 7.6 cm (3.0 in.) H2O.

**4.2.2** Level and zero the manometer. Check level at least once per hour. Record all necessary data on data sheet.

**4.2.3** Measure the velocity head and temperature at the traverse points. Ensure that the proper differential pressure gauge is being used for the range of dp values encountered. Conduct a post-sample leak-check as described in Section 4.2.1 above, to validate the traverse run. An initial velocity traverse will be conducted at each sampling location to support the selection of the appropriate sample nozzle for the modified Method 5 APFO isokinetic sampling method. Each of three APFO sample runs conducted at each location will include the determination of stack gas velocity and volumetric flow rate.

**4.2.4** Measure the static pressure in the stack. One reading is usually adequate per location.

**4.2.5** Determine the atmospheric pressure at the meter console location.

**4.2.6** Determine the stack gas dry molecular weight using the procedures described in Section 4.3. The team leader can assume a gas composition of 20.9% oxygen, 0.0% carbon dioxide and the balance nitrogen for non-combustion sources that utilize ambient air in the industrial process. The team leader can also assume a gas composition consistent with past experience on similar sources as an initialization parameter for the modified Method 5 APFO sampling method.

**4.2.7** Obtain the moisture content from using procedures described in Section 4.4. The team leader can use the alternative determination of moisture content using wet-bulb/dry-bulb temperature measurements as an initialization parameter for the modified Method 5 APFO sampling method.

**4.2.8** Determine the cross-sectional area of the stack or duct at the sampling location. Measure each diameter distance to verify its dimensions.

### **4.3 Gas Analysis for the Determination of Dry Molecular Weight**

**4.3.1** Perform the following procedures before measurement of emissions. An integrated multi-point sample of the stack gas will be taken concurrently with each modified Method 5 APFO sampling run. The integrated gas sample will either be analyzed for oxygen and carbon dioxide content in accordance with the procedures provided in EPA Method 3 or 3A found in 40 CFR Part 60 Appendix A. The team leader may assume the dry molecular weight of ambient air for ambient sources, confirmed by the use of a field portable oxygen analyzer, and forgo the collection of the integrated gas sample.

### **4.3.2 Multi-Point, Integrated Sampling Procedure.**

**4.3.2.1** Unless otherwise specified in an applicable regulation, or by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of 12 traverse points shall be used for all other cases. The traverse points shall be located according to EPA Method 1.

**4.3.2.2** Evacuate the flexible bag. Connect the probe, and place it in the stack, with the tip of the probe positioned at the sampling point. Purge the sampling line. Next, connect the bag, and make sure that all connections are tight.

**4.3.2.3** Sample Collection. Sample a slip stream at a constant rate from the exhaust of the modified EPA Method 5 APFO sample train ( $\pm 10$  percent). The sampling run should be simultaneous with, and for the same total length of time as, the APFO emission rate determination. Collection of at least 28 liters (1.0 ft<sup>3</sup>) of sample gas is recommended; however, smaller volumes may be collected, if desired.

**4.3.2.4** Obtain one integrated flue gas sample during each pollutant emission rate determination. Within 8 hours after the sample is taken, analyze it for percent CO<sub>2</sub> and percent O<sub>2</sub> using either an Orsat analyzer or instrumental analyzers.

**4.3.3** Integrated Sample Analysis using Orsat Analyzer. Use an Orsat analyzer to measure O<sub>2</sub> and CO<sub>2</sub> concentration for dry molecular weight determination, using procedures as specified in the analyzer user's manual. If an Orsat analyzer is used, it is recommended that the Orsat leak-check, described in Section 4.3.3.1, be performed before this determination; however, the check is optional. Calculate the dry molecular weight as indicated in Section 4.3.5. Repeat the analysis and calculation procedures until the individual dry molecular weights for any three analyses differ from their mean by no more than 0.3 g/g-mole (0.3 lb/lb-mole). Average these three molecular weights, and report the results to the nearest 0.1 g/g-mole (0.1 lb/lb-mole).

**4.3.3.1** Leak-Check Procedure for Orsat Analyzer. Moving an Orsat analyzer frequently causes it to leak. Therefore, an Orsat analyzer should be thoroughly leak-checked on site before the flue gas sample is introduced into it. The procedure for leak-checking an Orsat analyzer is as follows:

**4.3.3.2** Bring the liquid level in each pipette up to the reference mark on the capillary tubing, and then close the pipette stopcock.



**4.3.3.3** Raise the leveling bulb sufficiently to bring the confining liquid meniscus onto the graduated portion of the burette, and then close the manifold stopcock.

**4.3.3.4** Record the meniscus position.

**4.3.3.5** Observe the meniscus in the burette and the liquid level in the pipette for movement over the next 4 minutes.

**4.3.3.6** For the Orsat analyzer to pass the leak-check, two conditions must be met:

**4.3.3.7** The liquid level in each pipette must not fall below the bottom of the capillary tubing during this 4-minute interval.

**4.3.3.8** The meniscus in the burette must not change by more than 0.2 ml during this 4-minute interval.

**4.3.3.9** If the analyzer fails the leak-check procedure, check all rubber connections and stopcocks to determine whether they might be the cause of the leak. Disassemble, clean, and regrease any leaking stopcocks. Replace leaking rubber connections. After the analyzer is reassembled, repeat the leak-check procedure.

#### **4.3.4 Integrated Sample Analysis using Instrumental Analyzers**

**4.3.4.1** This is a modification of EPA Method 3A (40 CFR Part 60 Appendix A) for the analysis of an integrated gas sample (Tedlar Bags), where Method 3A is written for the continuous analysis of a sample gas stream over the duration of each sample run, and can be utilized at the discretion of the team leader. The dry stack gas will be analyzed with a Servomex 1440 oxygen and carbon dioxide analyzers calibrated to EPA Method 3A/7E specifications. Ambient air may be used for the oxygen span gas upper value. CEMS grade nitrogen will be used to zero instrumentation. All other calibration gases used will be EPA Protocol certified gases.

**4.3.4.2** Measurement System Preparation, Analyzer Calibration Error, Follow Sections 8.2 through 8.5 of EPA Method 7E to calibrate the analyzers directly with appropriate ranges of calibration gases. Record instrument information, date, operator and calibration information on Modified EPA Method 3A form. Calibration will consist of a zero gas (CEM grade nitrogen, upper span gas and mid-range gas. Zero instruments with nitrogen, set span to accurately read upper span gas, then introduce mid gas. All results of the linearization must be within 2% of span (range) of target concentration.

**4.3.4.3** Connect integrated bag sample to analyzer inlet and record to the tenth of a percent the stable values on Modified 3A data form for oxygen and carbon dioxide. One stable reading per integrated bag.

**4.3.4.4** Zero and Calibration Drift Test. Follow Section 8.5 of EPA Method 7E. Once all samples have been analyzed and recorded, repeat the analyzer linearization calibration error without adjustments to determine if analyzer drift has occurred. Drift of no more than 3% of span value will be accepted. Given the relatively short duration of time from initial linearization to final calibration this is not expected to be a concern.

**4.3.4.5** Emission Calculation. Measured concentrations of oxygen and carbon dioxide will be corrected for any calibration error or drift bias using the pre and post analysis analyzer calibration values for zero and upscale gas that is closest to the stack value.

**4.3.4.5.1** For all CO<sub>2</sub> analyzers, and for O<sub>2</sub> analyzers that can be calibrated with zero gas, express all concentrations as percent.

**4.3.4.5.2** For O<sub>2</sub> analyzers that use a low-level calibration gas in place of a zero gas, calculate the effluent gas concentration using the following equation.

$$C_{\text{gas}} = \frac{C_{\text{ma}} - C_{\text{oa}}}{C_{\text{m}} - C_{\text{o}}} (C_{\text{avg}} - c_{\text{m}}) + c_{\text{ma}}$$

Where:

- $C_{\text{gas}}$  = Effluent gas concentration, dry basis, percent.
- $C_{\text{ma}}$  = Actual concentration of the upscale calibration gas, percent.
- $C_{\text{oa}}$  = Actual concentration of the low-level calibration gas, percent.
- $C_{\text{m}}$  = Average of initial and final analyzer calibration bias check responses for the upscale calibration gas, percent.
- $C_{\text{o}}$  = Average of initial and final analyzer calibration bias check responses for the low level gas, percent.
- $C_{\text{avg}}$  = Average gas concentration indicated by the gas analyzer, dry basis, percent.

#### **4.3.5 Calculations and Data Analysis.**

**4.3.5.1** Nomenclature.

- $M_{\text{d}}$  = Dry molecular weight, g/g-mole (lb/lb-mole).

$\%CO_2$  = Percent  $CO_2$  by volume, dry basis.  
 $\%O_2$  = Percent  $O_2$  by volume, dry basis.  
 $\%CO$  = Percent  $CO$  by volume, dry basis.  
 $\%N_2$  = Percent  $N_2$  by volume, dry basis.  
0.280 = Molecular weight of  $N_2$  or  $CO$ , divided by 100.  
0.320 = Molecular weight of  $O_2$  divided by 100.  
0.440 = Molecular weight of  $CO_2$  divided by 100.

**4.3.5.2** Nitrogen, Carbon Monoxide Concentration. Determine the percentage of the gas that is  $N_2$  and  $CO$  by subtracting the sum of the percent  $CO_2$  and percent  $O_2$  from 100 percent.

**4.3.5.3** Dry Molecular Weight. Use the equation 3-1 of EPA Method 3, provided below, to calculate the dry molecular weight of the stack gas.

$$M_d = 0.440 (\%CO_2) + 0.320 (\%O_2) + 0.280 (\%N_2 + \%CO) \quad \text{Eq. 3-1}$$

## 4.4 Determination of Moisture Content in Stack Gases

EPA Method 4 (40 CFR Part 60 Appendix A) procedures will be utilized to determine the stack gas moisture content concurrent with each modified Method 5 APFO sampling run. The team leader can use the alternative determination of moisture content using wet- bulb/dry-bulb temperature measurements as an initialization parameter for the modified Method 5 APFO sampling method.

### 4.4.1 Preparation of Sampling Train

**4.4.1.1** Place known volumes of reagents in the impingers as described in Section 4.5, Determination of APFO Emissions from Stationary Sources. Weigh and record the weight of the desiccant impinger to the nearest 0.5 g.

**4.4.1.2** Set up the sampling train as shown in Figure 4-1 of EPA Method 4 and specified in Section 4.5. Turn on the probe heater and (if applicable) the filter heating system to temperatures of approximately  $120^\circ C$  ( $248^\circ F$ ), to prevent water condensation ahead of the condenser system. Allow time for the temperatures to stabilize. Place crushed ice and water in the ice bath container.

**4.4.1.3** Metering System. Same as that described in Section 4.5.

**4.4.1.4** Leak check: Plug the nozzle and pull a 380 mm (15 in.) Hg vacuum. A leakage rate in excess of 4 percent of the average sampling rate or 0.00057 m<sup>3</sup>/min (0.020 cfm), whichever is less, is unacceptable. Following the leak check, reconnect the probe to the sampling train.

**4.4.1.5** Sampling Train Operation. During the sampling run, maintain an isokinetic sampling rate consistent with the method for the Determination of APFO Emissions from Stationary Sources. For each run, record the data required on a data sheet. Be sure to record the dry gas meter reading at the beginning and end of each sampling time increment and whenever sampling is halted. Take other appropriate readings at each sample point at least once during each time increment.

**4.4.1.6** To begin sampling, position the probe tip at the first traverse point. Immediately start the pump, and adjust the flow to the desired rate. Traverse the cross section, sampling at each traverse point for an equal length of time. Add more ice and, if necessary, salt to maintain a temperature of less than 20 °C (68 °F) at the desiccant impinger outlet.

**4.4.1.7** After collecting the sample, conduct a leak check of the sampling train as described in Section 4.4.1.4. Record the leak rate. If the leakage rate exceeds the allowable rate, either reject the results or correct the sample volume as in Section 12.3 of EPA Method 5.

#### **4.4.2 Quality Control**

Section	Quality Control Measure	Effect
Section 4.4.1.5	Leak rate of the sampling system cannot exceed 4 percent of the average sampling rate or 0.00057 m <sup>3</sup> /min (0.020 cfm).	Ensures the accuracy of the volume of gas sampled (Reference Method).
Section 4.4.1.7	Leak rate of the sampling system cannot exceed 2 percent of the average sampling rate.	Ensures the accuracy of the volume of gas sampled (Approximation Method).

#### **4.4.3 Calibration and Standardization.**

NOTE: Maintain a laboratory log of all calibrations.

**4.4.3.1** Calibrate the metering system, temperature sensors, and barometer according to Sections 4.5.10.2.

#### 4.4.4 Analytical Procedure.

**4.4.4.1** Measure the volume of the moisture condensed in each of the impingers to the nearest ml. Determine the increase in weight of the desiccant plus impinger to the nearest 0.5 g. Record this information and calculate the moisture content, as described in Section 4.4.5.

#### 4.4.5 Data Analysis and Calculations.

Carry out the following calculations, retaining at least one extra significant figure beyond that of the acquired data. Round off figures after final calculation.

##### 4.4.5.1 Nomenclature

$B_{ws}$	=	Proportion of water vapor, by volume, in the gas stream.
$M_w$	=	Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).
$P_m$	=	Absolute pressure (for this method, same as barometric pressure) at the dry gas meter, mm Hg (in. Hg).
$P_{std}$	=	Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
$R$	=	Ideal gas constant, 0.06236 (mm Hg)(m <sup>3</sup> )/(g-mole)(K) for metric units and 21.85 (in. Hg)(ft <sup>3</sup> )/(lb-mole)(R) for English units.
$T_m$	=	Absolute temperature at meter, °K (°R).
$T_{std}$	=	Standard absolute temperature, 293 °K (528 °R).
$V_f$	=	Final volume of condenser water, ml.
$V_i$	=	Initial volume, if any, of condenser water, ml.
$V_m$	=	Dry gas volume measured by dry gas meter, dcm (dcf).
$V_{m(std)}$	=	Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm (dscf).
$V_{wc(std)}$	=	Volume of water vapor condensed, corrected to standard conditions, scm (scf).
$V_{wsg(std)}$	=	Volume of water vapor collected in silica gel, corrected to standard conditions, scm (scf).
$W_f$	=	Final weight of silica gel or silica gel plus impinger, g.
$W_i$	=	Initial weight of silica gel or silica gel plus impinger, g.
$Y$	=	Dry gas meter calibration factor.
$V_m$	=	Incremental dry gas volume measured by dry gas meter at each traverse point, dcm (dcf).
$\rho_w$	=	Density of water, 0.9982 g/ml (0.002201 lb/ml).

#### 4.4.5.2 Volume of Water Vapor Condensed

$$V_{wc(std)} = \frac{V_f - V_i}{(\rho)_{std}} \frac{R(T)_{std}}{M_w}$$

$$V_{wc(std)} = K_I (V_f - V_i)$$

Where:

$$\begin{aligned} K1 &= 0.001333 \text{ m}^3/\text{ml} \text{ for metric units,} \\ &= 0.04706 \text{ ft}^3/\text{ml} \text{ for English units.} \end{aligned}$$

#### 4.4.5.3 Volume of Water Collected in Silica Gel.

$$V_{wsg(std)} = \frac{(W_F - W_i)RT_{std}}{\rho_{std} M_w K_2} V_{wsg(std)} = K_3 (W_F - W_i)$$

where:

$$\begin{aligned} K2 &= 1.0 \text{ g/g for metric units} \\ &= 453.6 \text{ g/lb for English units} \\ K3 &= 0.001335 \text{ m}^3/\text{g for metric units,} \\ &= 0.04715 \text{ ft}^3/\text{g for English units.} \end{aligned}$$

#### 4.4.5.4 Sample Gas Volume

$$V_{m(std)} = \frac{V_m Y \rho_m T_{std}}{P_{std} T_m} = K4 Y \frac{V_m \rho_m}{T_m}$$

where:

$$\begin{aligned} K4 &= 0.3855 \text{ }^\circ\text{K/mm Hg for metric units,} \\ &= 17.64 \text{ }^\circ\text{R/in. Hg for English units.} \end{aligned}$$

NOTE: If the post-sample leak rate (Section 4.4.1.7) exceeds the allowable rate, correct the value of  $V_m$  in Equation 4-3, as described in Section 12.3 of Method 5.



#### 4.4.5.5 Moisture Content

$$B_{ws} = \frac{V_{wc(std)} + V_{wsg(std)}}{V_{wc(std)} + V_{wsg(std)} + V_{m(std)}}$$

**4.4.5.6** In saturated or moisture droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one using a value based upon the saturated conditions (see EPA Method 4 Section 4.1), and another based upon the results of the impinger analysis. The lower of these two values of  $B_{ws}$  shall be considered correct.

### 4.5 Determination of APFO Emissions from Stationary Sources

#### 4.5.1 Presampling Preparation

It is suggested that sampling equipment be maintained according to the procedures described in APTD-0576. The determination of APFO emissions from a stationary source includes the performance of three replicate sample runs conducted consecutively.

**4.5.1.1** Place 200 to 300 g of desiccant in each of several air-tight containers. Weigh each container, including desiccant, to the nearest 0.5 g, and record this weight. As an alternative, the desiccant need not be preweighed, but may be weighed directly in its impinger or sampling holder just prior to train assembly.

**4.5.1.2** Check filters visually against light for irregularities, flaws, or pinhole leaks. Label filters of the proper diameter on the back side near the edge using numbering machine ink. As an alternative, label the shipping containers (glass petri dishes), and keep each filter in its identified container at all times except during sampling.

#### 4.5.2 Preliminary Determinations.

**4.5.2.1** Select the sampling site and the minimum number of sampling points according to Section 4.5.2.2 Determine the stack pressure, temperature, and the range of velocity heads using Method 2; Determine the moisture content using Section 4.4. Determine the stack gas dry molecular weight, as described in Section 4.3.

**4.5.2.2** Select a nozzle size based on the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the

nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 8.3 of EPA Method 2).

**4.5.2.3** Select a suitable probe liner and probe length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the required probe length.

**4.5.2.4** Select a total sampling time greater than or equal to the minimum total sampling time specified in the procedures for the specific industry such that (1) the sampling time per point is not less than 2 minutes (or some greater time interval as specified by the Administrator), and (2) the sample volume taken (corrected to standard conditions) will exceed the required minimum total gas sample volume. The latter is based on an approximate average sampling rate. It is anticipated that APFO sample runs will be conducted over a 2-hour time period with a designed test volume between 60 and 90 dry standard cubic feet. The team leader is responsible for setting the test time and air sample volume for each test location.

**4.5.2.5** The sampling time at each point shall be the same. It is recommended that the number of minutes sampled at each point be an integer or an integer plus one-half minute, in order to avoid timekeeping errors.

### **4.5.3 Preparation of Sampling Train**

**4.5.3.1** During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin. Place 100 ml of deionized water in the first impinger, place 100 ml of 0.1N NaOH in the second impinger, place 100 ml of 0.01N  $\text{Na}_2\text{B}_4\text{O}_7$  buffer solution in the third impinger, add a preweighed desiccant impinger as the fourth impinger of the sample train.

**4.5.3.2** Using a tweezer or clean disposable surgical gloves, place a labeled (identified) filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed. Place the filter holder between last impinger and the silica gel impinger.

**4.5.3.3** When glass probe liners are used, install the selected nozzle using a silicone O- ring when stack temperatures are less than 260 °C (500 °F) or a heat-resistant graphite ferrule when temperatures are higher. When metal liners are used, install the nozzle as discussed above or by a leak-free direct mechanical connection. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

**4.5.3.4** Using a tweezer or clean disposable surgical gloves, place a labeled glass fiber filter (identified) in the filter holder to be placed in the oven. Be sure that the filter is properly centered and the gasket properly placed so as to prevent the sample gas stream from circumventing the filter. The filter support must be of non- fluoropolymer construction i.e silicone gasket glass frit. Check the filter for tears after assembly is completed.

**4.5.3.5** Attach to the oven filter outlet a coiled condenser in horizontal position to the top of XAD2 trap oriented vertically on top of a knock out impinger. The connection from the coiled condenser to the XAD2 trap should have a thermocouple in the gas stream to measure XAD2 trap temperature or exit of condenser. Alternatively, if necessary, tape a thermocouple to the exterior of the trap inlet and insulate well. Cover XAD2 trap to minimize light exposure. Connect submersible pump with tubing to deliver cooling water from the ice bath to the coiled condenser and jacketed XAD2 trap. This may be done in series with the coolest water entering the XAD2 trap bottom and exiting the coiled condenser at the inlet side.

**4.5.3.6** Set up the train as shown in Figure 5-1 of EPA Method 5, using a Silicone O-ring to achieve each ground glass joint seal.

**4.5.3.7** Place crushed ice around the impingers.

#### **4.5.4 Leak-Check Procedures.**

**4.5.4.1** Leak Check of Metering System Shown in Figure 5-1 of EPA Method 5. That portion of the sampling train from the pump to the orifice meter should be leak-checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. The following procedure is suggested (see Figure 5-2): Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13 to 18 cm (5 to 7 in.) water column by blowing into the rubber tubing. Pinch off the tubing, and observe the manometer for one minute. A loss of pressure on the manometer indicates a leak in the meter box; leaks, if present, must be corrected.

**4.5.4.2** Pretest Leak Check. A pretest leak check of the sampling train is recommended, but not required. If the pretest leak check is conducted, the following procedure should be used.

**4.5.4.3** After the sampling train has been assembled, turn on and set the probe heating systems to the desired operating temperatures. Allow time for the temperatures to stabilize. If a Viton A O-ring or

other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 380 mm (15 in.) Hg vacuum. Leakage rates in excess of 4 percent of the average sampling rate or 0.00057 m<sup>3</sup>/min (0.020 cfm), whichever is less, are unacceptable.

**NOTE:** A lower vacuum may be used, provided that it is not exceeded during the test.

**4.5.4.4** The following leak-check instructions for the sampling train described in APTD- 0576 and APTD-0581 may be helpful. Start the pump with the bypass valve fully open and the coarse adjust valve completely closed. Partially open the coarse adjust valve, and slowly close the bypass valve until the desired vacuum is reached. Do not reverse the direction of the bypass valve, as this will cause water to back up into the filter holder. If the desired vacuum is exceeded, either leak-check at this higher vacuum, or end the leak check and start over.

**4.5.4.5** When the leak check is completed, first slowly remove the plug from the inlet to the probe and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder and the desiccant from being entrained backward into the third impinge.

**4.5.4.6** Leak Checks During Sample Run. If, during the sampling run, a component (e.g., filter assembly or impinger) change becomes necessary, a leak check shall be conducted immediately before the change is made. The leak check shall be done according to the procedure outlined in Section 4.5.4.3 above, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the sample run. If the leakage rate is found to be no greater than 0.00057 m<sup>3</sup>/min (0.020 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered; if, however, a higher leakage rate is obtained, either record the leakage rate and plan to correct the sample volume as shown in Section 12.3 of this EPA Method 5, or void the sample run.

**NOTE:** Immediately after component changes, leak checks are optional. If such leak checks are done, the procedure outlined in Section 4.5.4.3 above should be used.

**4.5.4.7** Post-Sample Leak Check. A leak check of the sampling train is mandatory at the conclusion of each sampling run. The leak check shall be performed in accordance with the procedures outlined in Section 4.5.4.3, except that it shall be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is found to be no greater than

0.00057 m<sup>3</sup>/min (0.020 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, either record the leakage rate and correct the sample volume as shown in Section 12.3 of EPA Method 5, or void the sampling run.

#### **4.5.5 Sampling Train Operation.**

During the sampling run, maintain an isokinetic sampling rate (within 10 percent of true isokinetic unless otherwise specified by the Administrator) and a temperature in the sample probe of  $120 \pm 14$  °C ( $248 \pm 25$  °F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator. The sample filter is maintained at ambient temperature.

**4.5.5.1** For each run, record the data required on a data sheet such as the one shown in Figure 5-3 of EPA Method 5. Be sure to record the initial DGM reading. Record the DGM readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted. Take other readings indicated by Figure 5-3 at least once at each sample point during each time increment and additional readings when significant changes (20 percent variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

**4.5.5.2** Clean the portholes prior to the sample run to minimize the chance of collecting deposited material. To begin sampling, verify that the probe heating system is up to temperature, remove the nozzle cap, verify that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump, and adjust the flow to isokinetic conditions. Nomographs are available which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type S pitot tube coefficient ( $C_p$ ) is  $0.85 \pm 0.02$ , and the stack gas equivalent density [dry molecular weight ( $M_d$ )] is equal to  $29 \pm 4$ . APTD-0576 details the procedure for using the nomographs. A spreadsheet designed for EPA Method 5 calculations is the preferred means of calculating and adjusting the isokinetic sample rate.

**4.5.5.3** When the stack is under significant negative pressure (i.e., height of impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to prevent water from backing into the filter holder. If necessary, the pump may be turned on with the coarse adjust valve closed.

**4.5.5.4** When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

**4.5.5.5** Traverse the stack cross-section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes; this minimizes the chance of extracting deposited material.

**4.5.5.6** During the sample run, make periodic adjustments to keep the temperature of the sample probe at the proper level; add more ice and, if necessary, salt to maintain a temperature of less than 20°C (68°F) at the desiccant impinger outlet. Also, periodically check the level and zero of the manometer.

**4.5.5.7** If the pressure drop across the filter becomes too high, making isokinetic sampling difficult to maintain, the filter may be replaced in the midst of the sample run. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. Before a new filter assembly is installed, conduct a leak check (see Section 4.5.4.3). The total APFO weight shall include the summation of the filter assembly catches.

**4.5.5.8** A single train shall be used for the entire sample run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or in cases where equipment failure necessitates a change of trains. In all other situations, the use of two or more trains will be subject to the approval of the Administrator.

**NOTE:** When two or more trains are used, separate analyses of the front-half and (if applicable) impinger catches from each train shall be performed, unless identical nozzle sizes were used on all trains, in which case, the front-half catches from the individual trains may be combined (as may the impinger catches) and one analysis of front-half catch and one analysis of impinger catch may be performed. Consult with the Administrator for details concerning the calculation of results when two or more trains are used.

**4.5.5.9** At the end of the sample run, close the coarse adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final DGM meter reading, and conduct a post-sample leak check, as outlined in Section 4.5.4.3. Also, leak-check the pitot lines as described in EPA Method 2, Section 8.1. The lines must pass this leak check, in order to validate the velocity head data.

#### **4.5.6 Calculation of Percent Isokinetic.**

Calculate percent isokinetic to determine whether the run was valid or another sample run should be made. If there was difficulty in maintaining isokinetic rates because of source conditions, consult with the Administrator for possible variance on the isokinetic rates.

#### **4.5.7 Sample Recovery.**

**4.5.7.1** Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool.

**4.5.7.2** When the probe can be safely handled, wipe off all external PM near the tip of the probe nozzle, and place a cap over it to prevent losing or gaining PM. Do not cap off the probe tip tightly while the sampling train is cooling down. This would create a vacuum in the filter holder, thereby drawing water from the impingers into the filter holder.

**4.5.7.3** Before moving the sample train to the cleanup site, remove the probe from the sample train and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Remove the umbilical cord from the last impinger, and cap the impinger.

Disconnect the oven filter assembly, coiled condenser, XAD2 trap. If liquid is present in coiled condenser attempt to drain into trap before disconnecting. Cap all openings as disconnecting from the sample train. Either ground-glass stoppers, plastic caps, rubber stoppers or Para film may be used to close these openings.

**4.5.7.4** Transfer the probe oven filter assembly, coiled condenser, XAD2 trap and impinger assemblies to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

**4.5.7.5** Save a portion of the methanol used for cleanup as a blank. Take 200 ml of this methanol directly from the wash bottle being used, and place it in a polyethylene sample container labeled "methanol blank."

**4.5.7.6** Inspect the train prior to and during disassembly, and note any abnormal conditions. Treat the samples as follows:

**4.5.7.6.1** Container No. 1. Carefully remove the oven filter from the filter holder, and place it in an identified petri dish container or other container provided by the analytical laboratory.



Use a pair of tweezers and/or clean disposable surgical gloves to handle the filter. If it is necessary to fold the filter, do so such that the PM cake is inside the fold. Using a dry Nylon bristle brush and/or a sharp-edged blade, carefully transfer to the petri dish any PM and/or filter fibers that adhere to the filter holder gasket.

**4.5.7.6.2** Container No. 2. Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover PM or any condensate from the probe nozzle, probe fitting, probe liner, and filter glassware by washing these components with methanol and placing the wash in a polyethylene sample container. Deionized water may be used instead of methanol at the discretion of the team leader if it is found to be a more effective recovery solvent. In these cases, save a water blank.

Perform the sample recovery as follows:

**4.5.7.6.2.1** Carefully remove the probe nozzle. Clean the inside surface by rinsing with methanol from a wash bottle and brushing with a Nylon bristle brush. Brush until the methanol rinse shows no visible particles, after which make a final rinse of the inside surface with methanol. Dry the sample nozzle with acetone prior to the next sample run and at the completion of sampling at each location.

**4.5.7.6.2.2** Rinse the probe liner with methanol by tilting and rotating the probe while squirting methanol into its upper end so that all inside surfaces will be wetted with methanol. Let the methanol drain from the lower end into an Erlenmeyer flask fitted with a ground glass socket attached to and compatible with the probe liner ball joint. Follow the methanol rinse with a probe brush. Hold the probe in an inclined position, squirt methanol into the upper end as the probe brush is being pushed with a twisting action through the probe; catching any methanol and particulate matter that is brushed from the probe. Run the brush through the probe three times or more until no visible PM is carried out with the methanol or until none remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times since metal probes have small crevices in which particulate matter can be entrapped. Rinse the brush with methanol, and quantitatively collect these washings in the sample container. After the brushing, make a final methanol rinse of the probe. Rinse the probe with acetone to aid drying prior to use in the next sample run and at the end of sampling at each location.

**4.5.7.6.2.3** Clean the inside of the front half and back half of the oven filter holder and connecting glassware by rubbing the surfaces with a Nylon bristle brush if needed to remove visible particulate and rinsing with methanol. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush (if used) and filter holder. Carefully rinse out the back half of the filter holder also.

**4.5.7.6.3** Coiled condenser. Rinse the coiled condenser three times with methanol into Container No. 2.

**4.5.7.6.4** XAD2 trap. Seal both ends of the XAD2 trap and label for lab submittal.

**4.5.7.6.5** Impinger Contents. Treat the impingers as follows: Make a notation of any color or film in the liquid catch. Measure the liquid by the volumetric method or by the mass method. Method 5 allows for either approach. The volumetric method includes measuring the volumes of the XAD2 knockout impinger and the following three impingers individually with polyethylene graduated cylinders dedicated to each impinger reagent type to within 1 ml. Record the volume of liquid present. The mass method includes measuring the net change in mass of the knockout/impinger or the sample bottle. The mass method will be used whenever practical because it involves less sample transfers and fewer chances for cross contamination. The volume in each impinger is required to calculate the moisture content of the effluent gas. The contents of the knockout impinger (below XAD2 trap) is transferred to a polyethylene sample container (Container No. 3) labeled "deionized water". The contents of the first impinger after the knockout impinger is also transferred to the polyethylene sample container (Container No. 3) labeled "deionized water". Rinse the knockout and first impinger three times with methanol from a wash bottle and add the methanol wash to Container No. 2. Transfer the contents of the second impinger after the knockout to a polyethylene sample container (Container No. 4) labeled "0.1N NaOH". Rinse the impinger three times with methanol from a wash bottle and add the methanol wash to Container No. 2. Transfer the contents of the third impinger to a polyethylene sample container (Container No. 5) labeled "0.01N Buffer Solution". Rinse the impinger three times with methanol from a wash bottle and add the methanol wash to Container No. 2.

**4.5.7.6.6** Container No. 6. Carefully remove the final filter (after last impinger) from the filter holder, and place it in an identified petri dish container or other container provided by the analytical laboratory. Use a pair of tweezers and/or clean disposable surgical gloves to handle the filter. Using a dry Nylon bristle brush and/or a sharp-edged blade, carefully transfer to the petri dish any PM and/or filter fibers that adhere to the filter holder gasket. Clean the inside of the front

half and back half of the filter holder and connecting glassware by rubbing the surfaces with a Nylon bristle brush if needed to remove visible particulate and rinsing with methanol. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush (if used) and filter holder. Place the methanol rinses into container No. 2. After all methanol washings and particulate matter have been collected in the sample containers, tighten the lids on the all sample containers so that methanol will not leak out when it is shipped to the laboratory. Mark the height of the fluid levels to allow determination of whether leakage occurred during transport. Label the container to identify clearly its contents.

#### **4.5.8 Sample Transport.**

Whenever possible, containers should be shipped in such a way that they remain upright at all times. Samples to be stored and transported cooled

#### **4.5.9 Quality Control.**

Quality Control Measure	Effect
Sampling equipment leak check and calibration.	Ensures accurate measurement of stack gas flow rate, sample volume.

#### **4.5.10 Calibration and Standardization.**

NOTE: Maintain a laboratory log of all calibrations.

**4.5.10.1 Probe Nozzle.** Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the ID of the nozzle to the nearest 0.025 mm (0.001 in.). Make three separate measurements using different diameters each time, and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.). When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use.

**4.5.10.2 Pitot Tube.** Pitot tubes will be calibrated annually using the geometric criteria for design of "S" Type pitot tubes. Pitot tubes meeting the criteria will be assigned the coefficient of 0.84. Pitots tubes not meeting the criteria will be repaired to meet criteria or discarded. Prior to each test the pitot should be inspected for damage or misshapen openings.

#### **4.5.10.3 Metering System.**

**4.5.10.3.1 Calibration Prior to Use.** Before its initial use in the field, the metering system shall be calibrated as follows: Connect the metering system inlet to the outlet of a wet test meter

that is accurate to within 1 percent. The wet test meter should have a capacity of 30 liters/rev (1 ft<sup>3</sup>/rev). A spirometer of 400 liters (14 ft<sup>3</sup>) or more capacity, or equivalent, may be used for this calibration, although a wet test meter is usually more practical. The wet test meter should be periodically calibrated with a spirometer or a liquid displacement meter to ensure the accuracy of the wet test meter. Spirometers or wet test meters of other sizes may be used, provided that the specified accuracies of the procedure are maintained. Run the metering system pump for about 15 minutes with the orifice manometer indicating a median reading as expected in field use to allow the pump to warm up and to permit the interior surface of the wet test meter to be thoroughly wetted. Then, at each of a minimum of three orifice manometer settings, pass an exact quantity of gas through the wet test meter and note the gas volume indicated by the DGM. Also note the barometric pressure and the temperatures of the wet test meter, the inlet of the DGM, and the outlet of the DGM. Select the highest and lowest orifice settings to bracket the expected field operating range of the orifice. Use a minimum volume of 0.14 m<sup>3</sup> (5 ft<sup>3</sup>) at all orifice settings. Record all the data on a form similar to Figure 5-5 of EPA Method 5 field and calculate Y, the DGM calibration factor, and H@, the orifice calibration factor, at each orifice setting as shown on Figure 5-5. Allowable tolerances for individual Y and H@ values are given in Figure 5-5. Use the average of the Y values in the calculations in Section 4.5.

**4.5.10.3.1.1** Before calibrating the metering system, a leak check should be conducted. For metering systems having diaphragm pumps, the normal leak-check procedure will not detect leakages within the pump. For these cases the following leak-check procedure is suggested: make a 10-minute calibration run at 0.00057 m<sup>3</sup>/min (0.020 cfm). At the end of the run, take the difference of the measured wet test meter and DGM volumes. Divide the difference by 10 to get the leak rate. The leak rate should not exceed 0.00057 m<sup>3</sup>/min (0.020 cfm).

**4.5.10.3.2 Calibration After Use.** After each field use, the calibration of the metering system shall be checked by performing three calibration runs at a single, intermediate orifice setting (based on the previous field sampling runs), with the vacuum set at the maximum value reached during the sample run series. To adjust the vacuum, insert a valve between the wet test meter and the inlet of the metering system. Calculate the average value of the DGM calibration factor. If the value has changed by more than 5 percent, recalibrate the meter over the full range of orifice settings, as detailed in Section 4.5.10.2.1. The Alternate procedure described in EPA Method 5 may also be used if approved by Administrator.

**4.5.10.3.3 Acceptable Variation in Calibration.** If the DGM coefficient values obtained before and after a sample run series differ by more than 5 percent, the sample run series shall either be voided, or calculations for the sample run series shall be performed using whichever meter coefficient value (i.e., before or after) gives the lower value of total sample volume.

**4.5.10.4 Probe Heater Calibration.** Use a heat source to generate air heated to selected temperatures that approximate those expected to occur in the sources to be sampled. Pass this air through the probe at a typical sample flow rate while measuring the probe inlet and outlet temperatures at various probe heater settings. For each air temperature generated, construct a graph of probe heating system setting versus probe outlet temperature. The procedure outlined in APTD-0576 can also be used. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used. Also, probes with outlet temperature monitoring capabilities do not require calibration.

NOTE: The probe heating system shall be calibrated before its initial use in the field.

**4.5.10.5 Temperature Sensors.** Use the procedure in Section 10.3 of EPA Method 2 to calibrate in-stack temperature sensors. Dial thermometers, such as are used for the DGM and condenser outlet, shall be calibrated against mercury-in-glass thermometers.

**4.5.10.6 Barometer.** Calibrate against a mercury barometer.

#### **4.5.11 Analytical Procedure.**

All analytical samples will be sent to an off-site analytical laboratory for analysis.

#### **4.5.12 Data Analysis and Calculations.**

Carry out calculations, retaining at least one extra significant figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used, provided that they give equivalent results.

##### **4.5.12.1 Nomenclature.**

An	=	Cross-sectional area of nozzle, m <sup>2</sup> (ft <sup>2</sup> ).
Bws	=	Water vapor in the gas stream, proportion by volume.
cAPFO	=	Concentration of APFO in stack gas, dry basis, corrected to standard conditions, lb/dscf.
EAPFO	=	Mass emission rate of APFO, lb/hr
I	=	Percent of isokinetic sampling.

mAPFO	=	Total amount of APFO collected, lb.
Mw	=	Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).
Pbar	=	Barometric pressure at the sampling site, mm Hg (in. Hg).
Ps	=	Absolute stack gas pressure, mm Hg (in. Hg).
Pstd	=	Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
R	=	Ideal gas constant, 0.06236 [(mm Hg)(m <sup>3</sup> )]/[(K)(g-mole)] {21.85 [(in. Hg)(ft <sup>3</sup> )]/[(°R)(lb-mole)]}.
Tm	=	Absolute average DGM temperature (see Figure 5-3), K (°R).
Ts	=	Absolute average stack gas temperature (see Figure 5-3), K (°R).
Tstd	=	Standard absolute temperature, 293 K (528 °R).
Vlc	=	Total volume of liquid collected in impingers and silica gel (see Figure 5-6), ml.
Vm	=	Volume of gas sample as measured by dry gas meter, dcm (dcf).
Vm(std)	=	Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscm (dscf).
Vw(std)	=	Volume of water vapor in the gas sample, corrected to standard conditions, scm (scf).
vs	=	Stack gas velocity, calculated by Method 2, Equation 2-7, using data obtained from Method 5, m/sec (ft/sec).
Qd	=	Stack gas volumetric flow rate, DSCFM
Y	=	Dry gas meter calibration factor.
.ΔH	=	Average pressure differential across the orifice meter (see Figure 5-4), mm H <sub>2</sub> O (in. H <sub>2</sub> O).
ø	=	Total sampling time, min.
dp	=	Sampling time interval, from the final (nth) component change until the end of the sampling run, min.
13.6	=	Specific gravity of mercury.
60	=	Sec/min.
100	=	Conversion to percent.

**4.5.12.2 Average Dry Gas Meter Temperature and Average Orifice Pressure Drop.** See data sheet (EPA Method 5 Figure 5-3).

**4.5.12.3 Dry Gas Volume.** Correct the sample volume measured by the dry gas meter to standard conditions (20 °C, 760 mm Hg or 68 °F, 29.92 in. Hg) by using the following equation:

$$V_{m(std)} V_m Y \frac{T_{std} \left( \rho_{bar} + \frac{\Delta H}{13.6} \right)}{T_m P_{std}} = K_i V_m Y \frac{\rho_{bar} + \frac{\Delta H}{13.6}}{T_m}$$

where:

- K1 = 0.3858 °K/mm Hg for metric units,  
 = 17.64 °R/in. Hg for English units.

#### 4.5.12.4 Volume of Water Vapor Condensed.

$$V_{w(std)} = \frac{\rho_w R T_{std}}{M_w P_{std}} = K_2 V_{lc}$$

where:

- K2 = 0.001333 m3/ml for metric units,  
 = 0.04706 ft3/ml for English units.

#### 4.5.12.5 Moisture Content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}}$$

NOTE: In saturated or water droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one from the impinger analysis (equation above), and a second from the assumption of saturated conditions. The lower of the two values of Bws shall be considered correct. The procedure for determining the moisture content based upon the assumption of saturated conditions is given in Section 4.0 of EPA Method 4. For the purposes of this method, the average stack gas temperature from each sampling run may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is  $\pm 1^\circ\text{C}$  ( $2^\circ\text{F}$ ).

**4.5.12.6 Total APFO Mass.** Determine the total collected APFO from the sum of the APFO mass obtained from the analysis of the contents sample containers 1 through 5.



#### 4.5.12.7 APFO Concentration.

$$C_{APFO} = m_{APFO} / V_m(\text{std})$$

#### 4.5.12.8 Isokinetic Variation.

$$I + \frac{100 T_s \left[ K_4 V_{lc} \frac{V_m Y}{T_m} \left( P_{bar} + \frac{\Delta H}{13.6} \right) \right]}{\theta v_s P_s A_n}$$

where:

$$\begin{aligned} K_4 &= 0.003454 [(\text{mm Hg})(\text{m}^3)]/[(\text{ml})(^\circ\text{K})] \text{ for metric units,} \\ &= 0.002669 [(\text{in. Hg})(\text{ft}^3)]/[(\text{ml})(^\circ\text{R})] \text{ for English units.} \end{aligned}$$

**4.5.12.9** Acceptable Results. If  $90 \% \leq I \leq 110 \%$ , the results are acceptable.

**4.5.12.10** Stack Gas Velocity and Volumetric Flow Rate. Calculate the average stack gas velocity and volumetric flow rate, if needed, using data obtained in this method and the equations in Sections 12.3 and 12.4 of EPA Method 2.

#### 4.5.12.11 APFO Mass Emission Rate

$$E_{APFO} = C_{APFO} \times Q_d \times 60$$

Figure 1 - Typical Glass Cloth Process Diagram

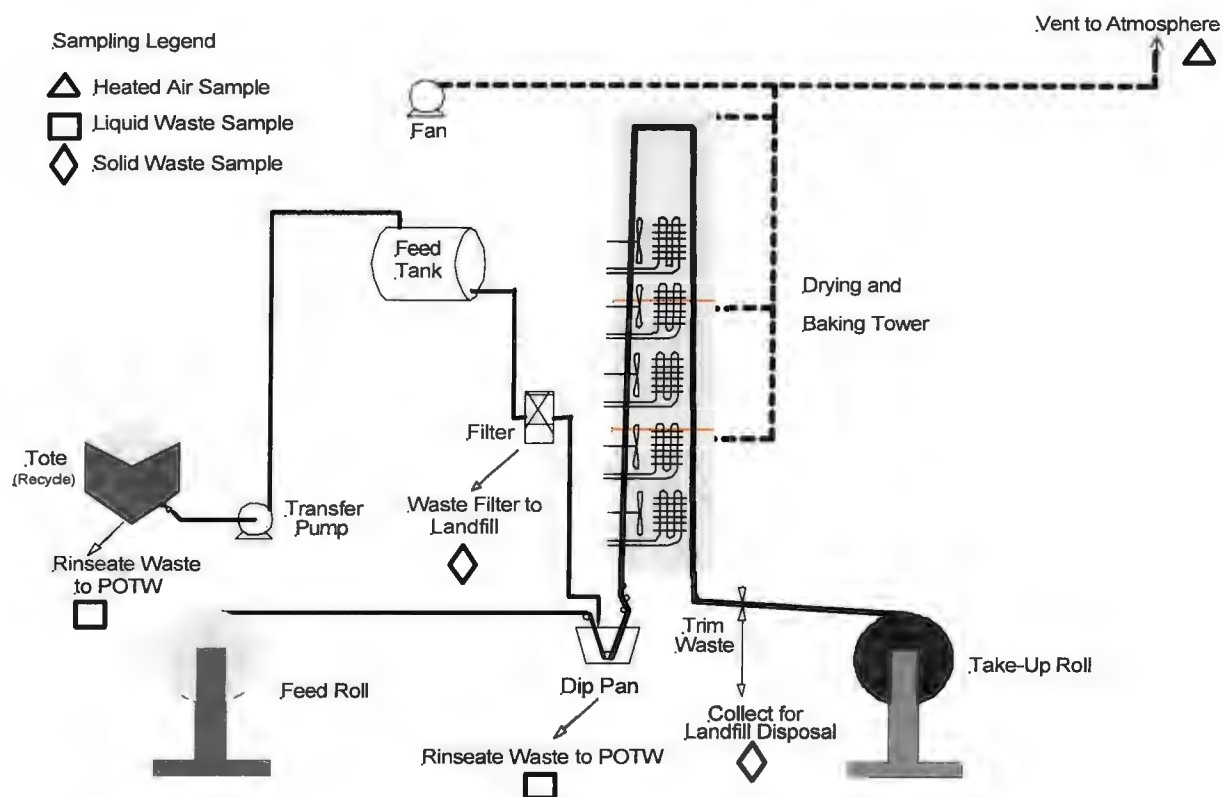
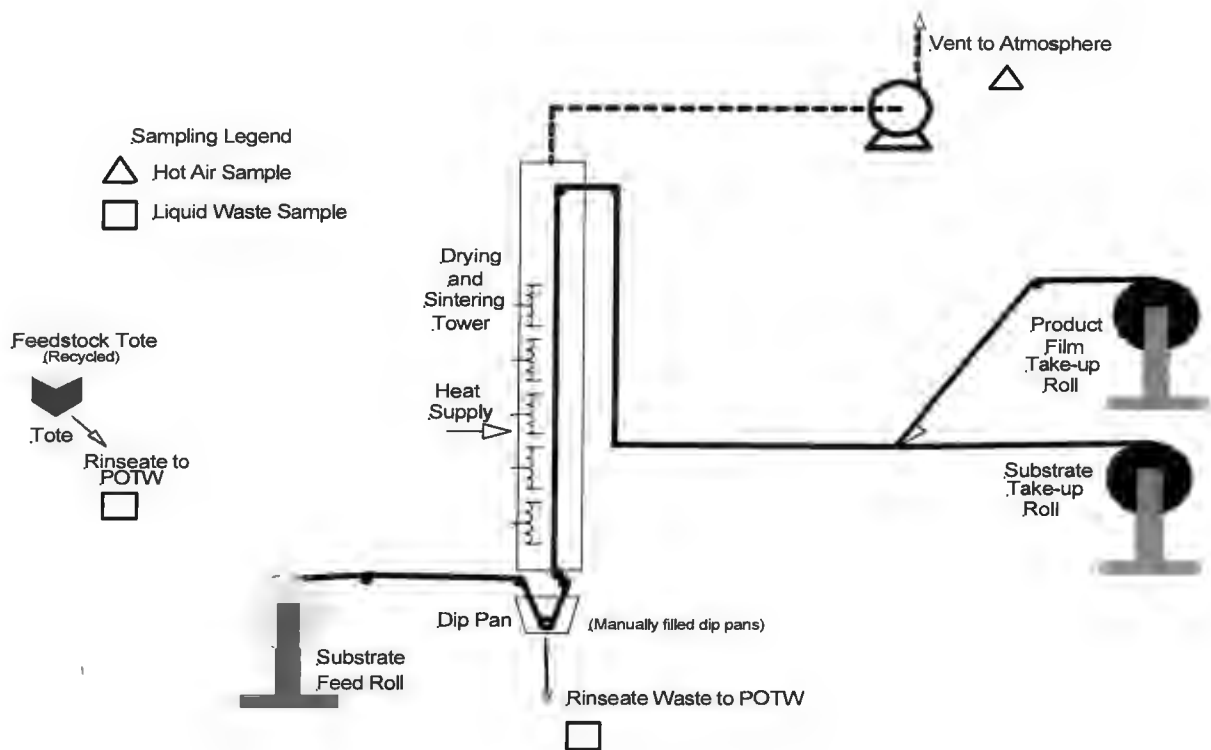


Figure 2 - Typical Cast Film Process Diagram



SAINT GOBAIN PERFORMANCE PLASTICS  
MERRIMACK, NEW HAMPSHIRE

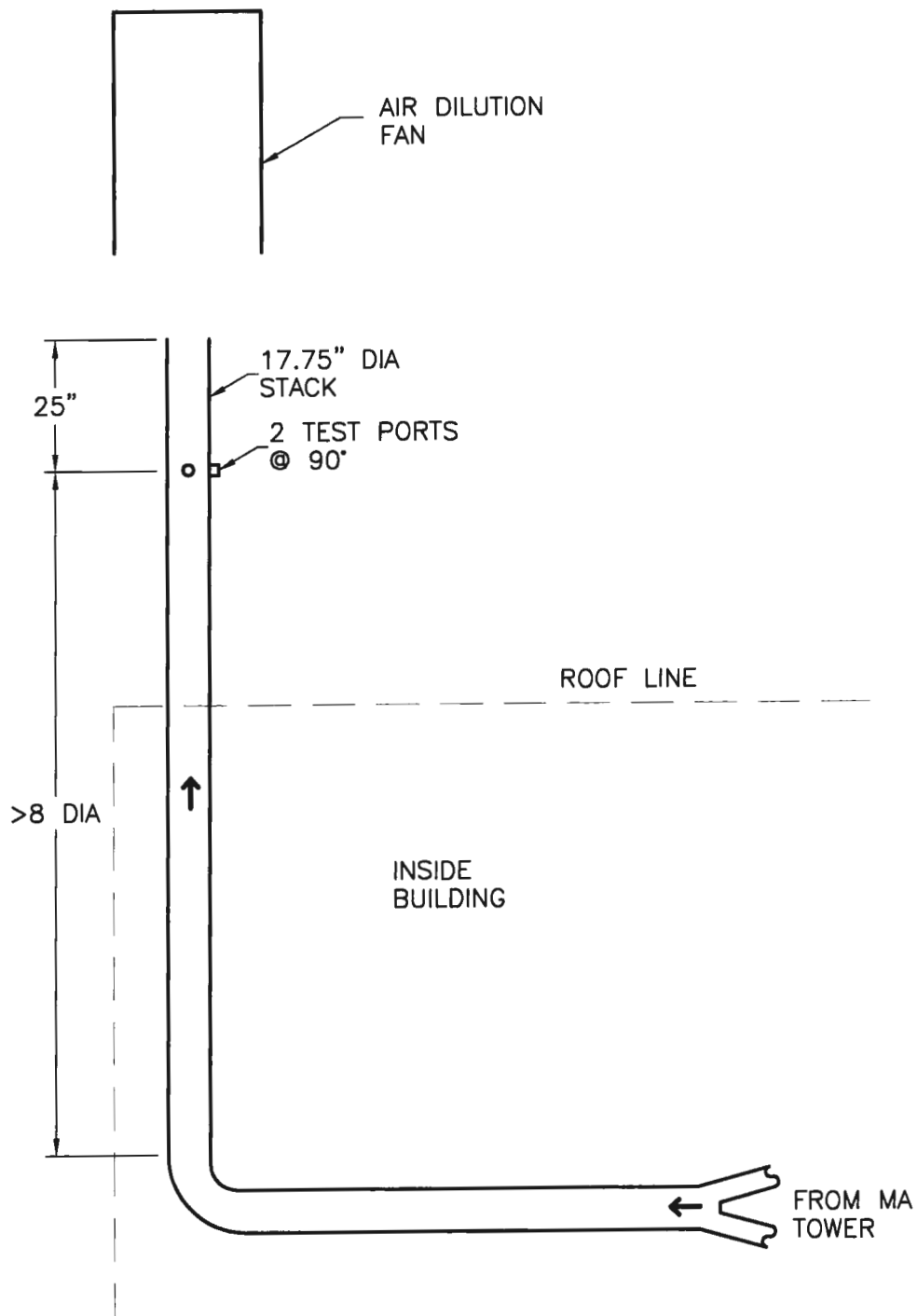


FIGURE 3  
TEST PORT LOCATIONS  
MA TOWER EXHAUST

NOT TO SCALE

SAINT GOBAIN PERFORMANCE PLASTICS  
MERRIMACK, NEW HAMPSHIRE

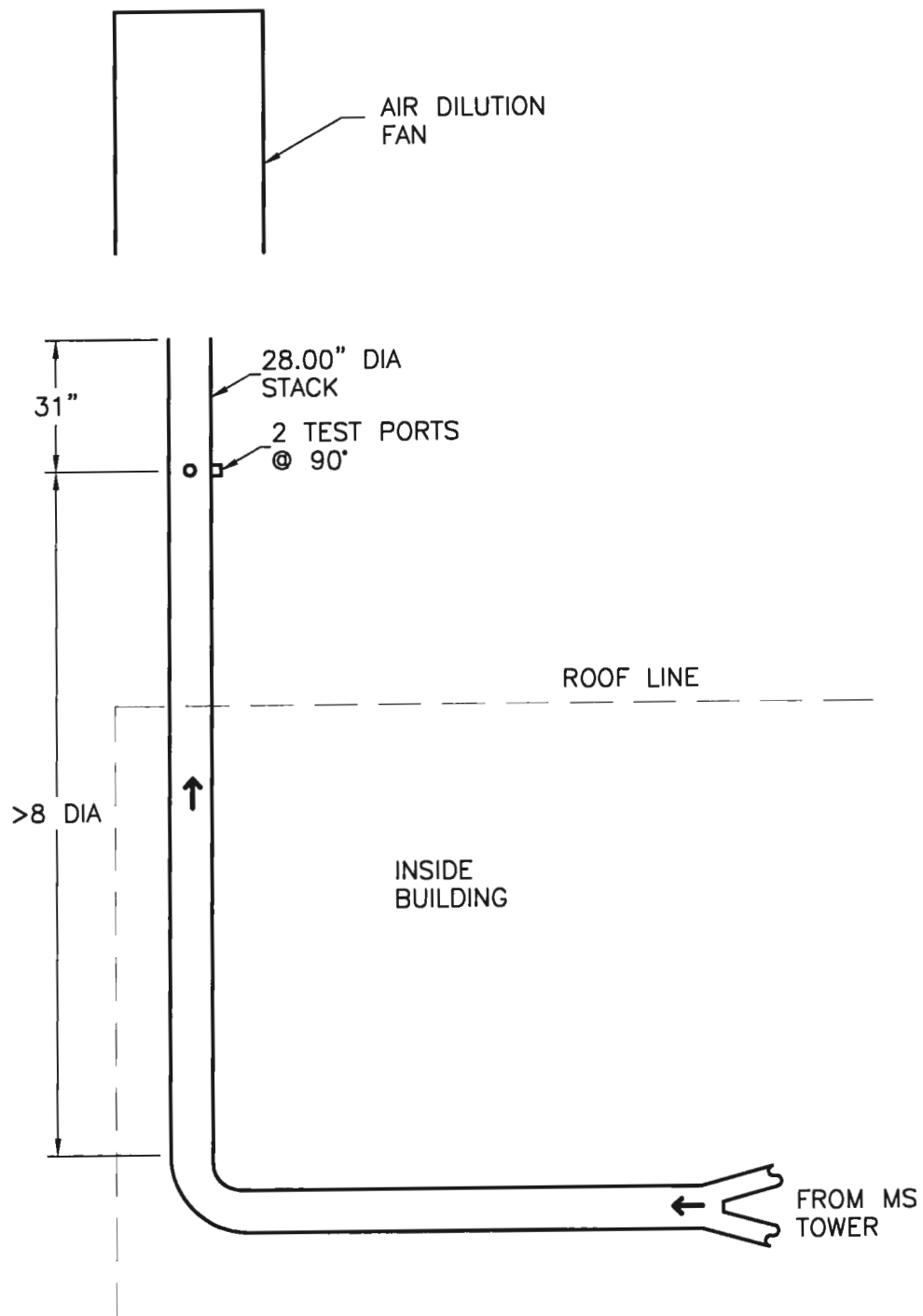


FIGURE 4  
TEST PORT LOCATIONS  
MS TOWER EXHAUST

NOT TO SCALE

SAINT GOBAIN PERFORMANCE PLASTICS  
MERRIMACK, NEW HAMPSHIRE

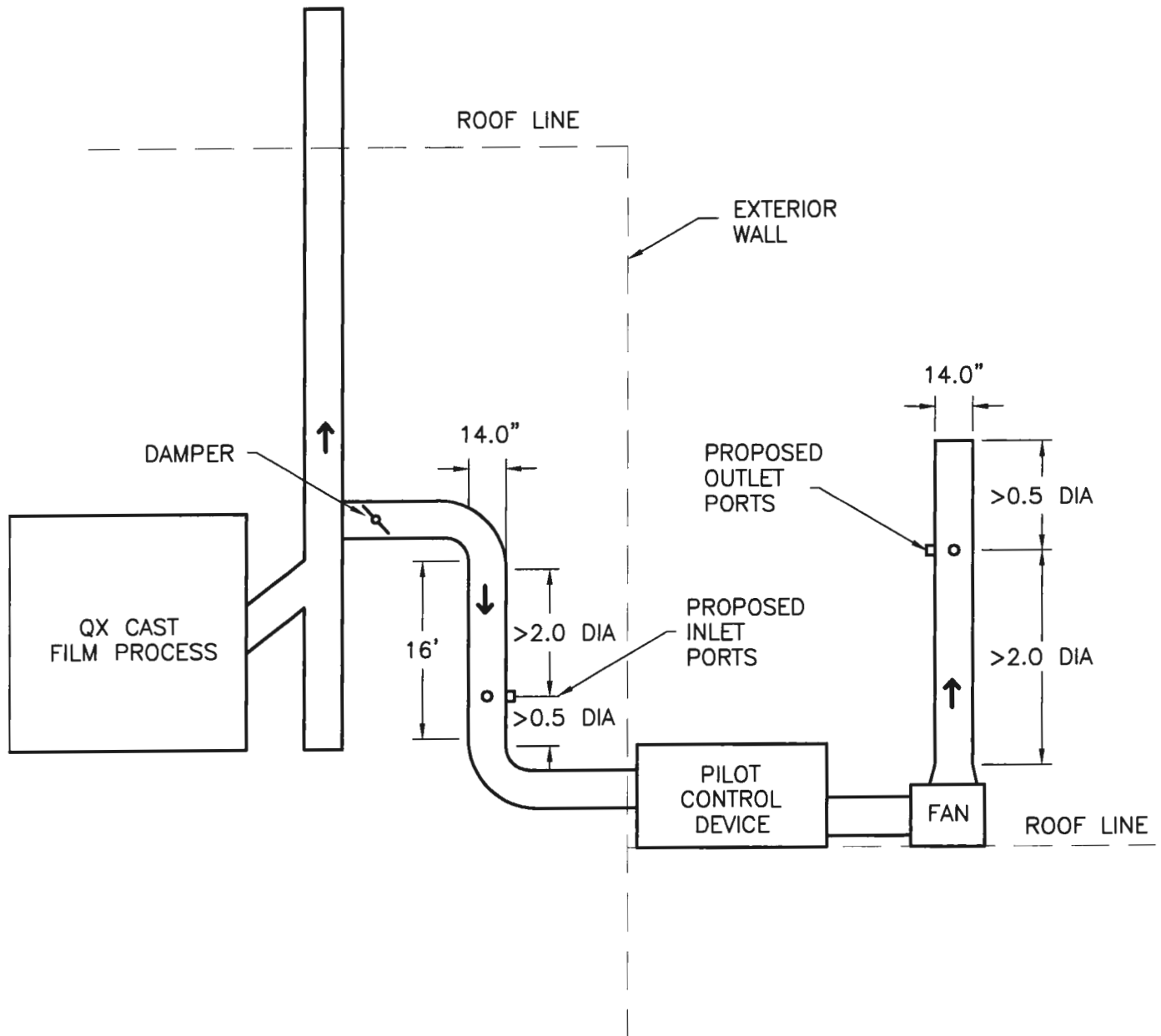


FIGURE 5  
TEST PORT LOCATIONS  
QX CAST FILM INLET/OUTLET

NOT TO SCALE

Report Of Analysis  
EPA Protocol Gas Mixtures

BARR01

REPORT NO: 63595-01

TO: Barr Engineering Co  
Attn: Benjamin Wiltse  
5150 West 76th Street  
Edina, MN 55439-2900  
(952) 832-2885

REPORT DATE: August 29, 2013

CUSTOMER PO NO: BAW06112013

CYLINDER NUMBER: CA03980

CYLINDER SIZE: 150A (141 std cu ft)  
CYLINDER PRESSURE: 2000 psig

COMPONENT	CONCENTRATION (v/v) ± EPA UNCERTAINTY	REFERENCE STANDARD		ANALYZER MAKE, MODEL, S/N, DETECTION	REPLICATE ANALYSIS DATA
Carbon dioxide	4.90 ± 0.06 %	GMIS	SRM 1674b	Varian Model 3400	<u>8/19/2013</u>
			Samp#: 7-14-E	Serial # 10680	4.90 %
		Cyl#: CC116770	Cyl#: CLM006389	Thermal Conductivity	4.90 %
		7.99 ± 0.08 %	6.98 ± 0.07 %	Gas Chromotography	4.89 %
		Exp: 8/2/2019	Exp: 6/16/2012	LAST CAL DATE: 8/7/2013	$\bar{x}$ : 4.90 %
Oxygen	21.23 ± 0.22 %	GMIS	SRM 2659a	Varian Model 3800	<u>8/26/2013</u>
			Samp#: 71-D-23	Serial # None	21.22 %
		Cyl#: CC88824	Cyl#: CAL015788	Thermal Conductivity	21.23 %
		24.92 ± 0.25 %	20.72 ± 0.043 %	Gas Chromotography	21.24 %
		Exp: 2/25/2021	Exp: 1/1/2016	LAST CAL DATE: 8/7/2013	$\bar{x}$ : 21.23 %

Nitrogen Balance

CERTIFICATION DATE: August 19, 2013

EPA EXPIRATION DATE: August 20, 2021

ppm =  $\mu$ mole/mole

% = mole-%

 $\bar{x}$  = EPA weighted mean

The above analyses were performed in accordance with Procedure G1 of the EPA Traceability Protocol, Report Number EPA600/R-12/531, dated May 2012.

The above analyses should not be used if the cylinder pressure is less than 100 psig.

ANALYST:

M.S. Calhoun

APPROVED:

J. T. Marrin

The only liability of this company for gas which fails to comply with this analysis shall be replacement or reanalysis thereof by the company without extra cost.



## EPA PROTOCOL GAS CERTIFICATE OF ANALYSIS

Cylinder Number: EB0099614  
Product ID Number: 126786  
Cylinder Pressure: 1900 PSIG  
COA #: EB0099614.20170726-0  
Customer PO. NO.:  
Customer:

Certification Date: 08/14/2017  
Expiration Date: 08/12/2025  
MFG Facility: - Shreveport - LA  
Lot Number: EB0099614.20170726  
Tracking Number: B1908036  
Previous Certification Dates:

This calibration standard has been certified per the May 2012 EPA Traceability Protocol, Document EPA-600/R-12/531, using procedure G1.

Do Not Use This Cylinder Below 100 psig (0.7 Megapascal).

### Certified Concentration(s)

Component	Concentration	Uncertainty	Analytical Principle	Assayed On
Carbon Dioxide	9.5 %	±0.10 %	NDIR	08/14/2017
Oxygen	9.46 %	±0.05 %	MPA	08/09/2017
Nitrogen	Balance			

Analytical Measurement Data Available Online.

### Reference Standard(s)

Serial Number	Lot	Expiration	Type	Balance	Component	Concentration	Uncertainty(%)	NIST Reference
EB0060740	EB0060740.20170209	08/05/2025	GMIS	N2	O2	24 %	0.502	071001
SG9916836	SG-9916836	06/06/2022	NTRM	N2	CO2	19.98 %	0.7	101001

### Analytical Instrumentation

Component	Principle	Make	Model	Serial	MPC Date
O2	MPA	Thermo	410i	1162980025	08/09/2017
CO2	NDIR	Thermo	410i	1162980025	08/14/2017

### SMART-CERT



This is to certify the gases referenced have been calibrated/tested, and verified to meet the defined specifications. This calibration/test was performed using Gases or Scales that are traceable through National Institute of Standards and Technology (NIST) to the International System of Units (SI). The basis of compliance stated is a comparison of the measurement parameters to the specified or required calibration/testing process. The expanded uncertainties use a coverage factor of k=2 to approximate the 95% confidence level of the measurement, unless otherwise noted. This calibration certificate applies only to the item described and shall not be reproduced other than in full, without written approval from Red Ball Technical Gas Services. If not included, the uncertainty of calibrations are available upon request and were taken into account when determining pass or fail.

*Brandon Theus*

Brandon Theus  
Analytical Chemist  
Assay Laboratory: Red Ball TGS  
Version 02-G, Revised on 2017-06-29



Assay Laboratory: Red Ball TGS  
555 Craig Kennedy Way  
Shreveport, LA 71107  
800-551-8150

## CERTIFICATE OF ANALYSIS (Zero Ambient Nitrogen)

Cylinder Number:	EB0099613	Certification Date:	07/27/2017
Product ID Number:	121026	Expiration Date:	07/25/2025
Cylinder Pressure:	1900 PSIG	MFG Facility:	RBTGS-Shreveport-LA
COA #	EB0099613.20170727-0	Lot Number:	EB0099613.20170727
Customer PO. NO.:		Tracking Number:	B1908012
Customer:		Previous Certification Dates:	

This mixture is for laboratory use only, not for drug, household or other use.

This mixture is certified in Mole % to be within  $\pm 2\%$  of the actual number reported with a confidence of 95%.

This mixture was manufactured by scale; weights traceable to N.I.S.T. Certificate #822/266926-02.

Do Not Use This Cylinder Below 100 psig (0.7 Megapascal).


Composing Material: Zero Ambient Nitrogen, Cert., Sz152

Component	Specification	Concentration
Nitrogen	Balance	Balance
Oxygen as Impurity	<1.0 PPM	<1.0 PPM
Carbon Dioxide as Impurity	<0.5 PPM	<0.5 PPM
Carbon Monoxide as Impurity	<0.5 PPM	<0.5 PPM
Total Oxides of Nitrogen as Impurity	<0.1 PPM	<0.1 PPM
Sulfur Dioxide as Impurity	<0.1 PPM	<0.1 PPM
Total Hydrocarbons as Impurity	<0.1 PPM	<0.1 PPM

Red Ball Technical Gas Service  
PGVP Vendor ID # G12017  
Information and Ordering  
800-551-8150  
Fax (318-425-6309)



PJLA  
Calibration and Testing  
Accreditation #62754

  
Anthony Cyr  
Analytical Chemist

Version 02-B, Revised on 2015-05-27

Appendix A-6  
ORD Report #1  
April 4, 2018



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
NATIONAL EXPOSURE RESEARCH LABORATORY

April 4, 2018

Mr. Clark Friese, Assistant Commissioner  
New Hampshire Department of Environmental Services (NH DES)  
29 Hazen Drive  
P.O. Box 95  
Concord, New Hampshire 03301

Dear Mr. Friese:

I am pleased to provide you with this initial laboratory report of perfluorocarboxylate (PFCA) concentrations in solid samples (char and soil). This report is in response to your request of June 22, 2017 asking for laboratory assistance analyzing per- and polyfluoroalkyl substances (PFAS) in environmental samples. PFCAs are a subset of PFAS. This report relates to solid matrix samples sent to our Athens Lab that included three tower char and another three soil samples. We understand that these samples were collected by New Hampshire Department of Environmental Services (NH DES), on August 23, 2017 and September 8, 2017, respectively. These samples were received at our Athens Lab by Dr. John Washington who was also responsible for their lab processing.

It is our understanding that this information was requested by NH DES to help in their ongoing investigation into the presence of PFAS in the environment near manufacturing facilities of interest. This request relates to our research capabilities and interests applying targeted and non-targeted analysis methods for discovery of the nature and extent of PFAS environmental occurrence associated with industrial releases. The current report is limited to targeted results only. Our non-targeted work requires considerable post-processing manual effort and therefore will lag the targeted results.

EPA continues to develop analytical methods for many PFAS compounds in various media including some of those included in this report. The data enclosed provides information related to the concentration of certain PFAS in the media sampled. In this report we do not interpret exposure or risk from these values. EPA does not currently have health based standards, toxicity factors or associated risk levels for PFAS, other than perfluorooctanoic acid (PFOA), perfluorooctane sulfonate (PFOS), and perfluorobutanesulfonic acid (PFBS). Therefore, while the data presented indicate the presence of PFCA, no conclusions can be made related to human or environmental exposure and risk.

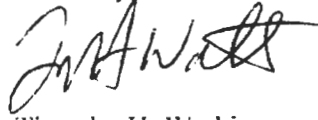
Thank you for inviting us to be part of this effort that helps to further both EPA's and New Hampshire's understanding of an important issue in the state. This is just one of many Agency efforts that demonstrate EPA's commitment to cooperative federalism.

April 4, 2017

The results presented in the attachment represent the work of many within ORD's National Exposure Research Laboratory. Our technical experts include Drs. John Washington, Mark Strynar, Andy Lindstrom, Seth Newton, Thomas Jenkins, and James McCord. Our Quality Assurance team includes Sania Tong-Argao and Brittany Stuart. Management support and coordination has been provided by Drs. Timothy Buckley, Myriam Medina-Vera, Jack Jones, Adam Biales, and Brian Schumacher.

If you have any questions or concerns, do not hesitate to contact me at (919) 541-2106 or via email at [watkins.tim@epa.gov](mailto:watkins.tim@epa.gov) or Tim Buckley at (919) 541-2454 or via email at [buckley.timothy@epa.gov](mailto:buckley.timothy@epa.gov). I look forward to our continued work together.

Sincerely,

A handwritten signature in black ink, appearing to read 'Tim H. Watkins', with a stylized flourish at the end.

Timothy H. Watkins  
Director

Attachment

cc: Meghan Cassidy, USEPA, Region 1  
Deb Szaro, USEPA, Region 1  
Jeff Morris, USEPA OPPT  
Betsy Behl, USEPA, OW  
Peter Grevatt, USEPA, OW  
Andy Gillespie, USEPA, ORD  
Timothy Buckley, USEPA, ORD

## Summary of Methods and Results

New Hampshire Department of Environmental Services (NH DES), in coordination with Region 1, requested ORD's technical support in analyzing PFAS in environmental samples potentially impacted by industrial sites within the state. NH DES assumed responsibility for the collection of samples and their shipment to our laboratories. ORD was responsible for sample extraction and analysis of PFAS. We are providing the results of our analysis as they become available. This is our first report.

The current report includes results for char (n=3) and soil (n=3) samples that were sent to and analyzed under the direction of Dr. John Washington within our Athens Lab. Samples were collected in containers provided by NH DES and shipped to EPA. Thirteen PFCA analytes (Table 1) were analyzed using methods described within an approved Quality Assurance Project Plan (QAPP)<sup>1</sup> and that have been generally described in Rankin et al., 2015.<sup>2</sup> In brief, each sample was divided into three ~1 g aliquots and extracted and analyzed in triplicate. Extracts were analyzed by liquid chromatography / mass spectrometry (Waters Acquity UPLC coupled to a Waters Quattro Premier XE tandem) and quantified using mass-labeled internal standards. These analyses were performed on samples, process blanks, and check standards using internal-calibration curves for quantitation. The mean value of the triplicate analysis is reported. Reported results are based on the identification and quantification of analytes using certified standards (i.e., targeted analysis).

Some of the sample extract required dilution so that concentrations were within the acceptable range of the calibration curve. The reported results have been adjusted for each dilution factor and flagged accordingly in Table 2.

**Table 1. Summary of Reported Perfluorocarboxylates**

<b>PFCA</b>	<b>Compound Name</b>	<b>Acronym</b>	<b>CAS Number</b>
C4	Perfluorobutanoic acid	PFBA	375-22-4
C5	Perfluoropentanoic acid	PFPeA	2706-90-3
C6	Perfluorohexanoic acid	PFHxA	307-24-4
C7	Perfluoroheptanoic acid	PFHpA	375-85-9
C8	Perfluorooctanoic acid	PFOA	335-67-1
C9	Perfluorononanoic acid	PFNA	375-95-1
C10	Perfluorodecanoic acid	PFDA	335-76-2
C11	Perfluoroundecanoic acid	PFUnDA	2058-94-8
C12	Perfluorododecanoic acid	PFDoDA	307-55-1
C13	Perfluorotridecanoic acid	PFTTrDA	72629-94-8

<sup>1</sup> Strynar, M.; Washington, J.; Lindstrom, A.; Henderson, W. 2017. Quality Assurance Project Plan: Non-Targeted Analyses of Per- and Polyfluoroalkyl Substances (PFAS) for New Hampshire Department of Environmental Services (NHDES). D-EMMD-PHCB-015-QAPP-01.

<sup>2</sup> K. Rankin, S. A. Mabury, T. M. Jenkins, J. W. Washington, A North American and global survey of perfluoroalkyl substances in surface soils: Distribution patterns and mode of occurrence. Chemosphere 161, 333-341 (2015).

C14	Perfluorotetradecanoic acid	PFTeDA	376-06-7
C16	Perfluorohexadecanoic acid	PFHxDA	67905-19-5
C18	Perfluorooctadecanoic acid	PFOcDA	16517-11-6

Targeted results are provided in Table 2 below for 13 perfluorocarboxylates that range from C4 through C18. Results are reported in mass of PFAS per unit mass of dry solid. Please note that the units are reported in  $\mu\text{g/g}$  for char samples and  $\text{pg/g}$  for soil samples. Precision of our measurements was estimated by the relative standard deviation (RSD) of triplicate extractions/analyses. For char across all of the compounds, the median RSD was 12.9% and ranged from 1.6% to 48.8%. Similarly for soils, the median RSD was 19.2% and ranged from 1.2 to 51.6%. The values reported for target analytes all exceeded levels detected in process blanks ( $p \leq 0.05$ ) and were corrected for any low detections in process blanks. No field blanks were provided or analyzed; however, quality control check standards were analyzed at varying concentrations throughout the analysis to ensure that measurements at varying points of the calibration range were within quality control specifications. Recovery of these standards ranged from 79.3% to 118% which was within our  $\pm 30\%$  criteria for acceptability.

As was expected, the measured perfluorocarboxylates tended to occur at higher concentrations in the char samples than the soil with char results ranging from <LOD (limit of detection) to 1430  $\mu\text{g/g}$ . Soil results were in the  $\text{pg/g}$  range and varied from <LOD to 7420  $\text{pg/g}$ . Among the char, PFCA concentrations were consistently higher at "QX Tower" followed by the "MS Tower," and lowest for the "MA Tower." Similarly for soil, PFCA concentrations generally trended "EPAORDS1" > "EPAORDS2" > "EPAORDS3." Across both matrices, the highest concentration was consistently observed for C8 (PFOA).

**Table 2. Concentration of Perfluorocarboxylates Measured in New Hampshire Samples**

PFCA	Sample ID	Matrix	Conc.	Unit	Flag(s)*
C4	MS Tower	Char	3.13	$\mu\text{g/g}$	D2
C5	MS Tower	Char	4.41	$\mu\text{g/g}$	D2
C6	MS Tower	Char	15.1	$\mu\text{g/g}$	D2
C7	MS Tower	Char	7.60	$\mu\text{g/g}$	D2
C8	MS Tower	Char	439	$\mu\text{g/g}$	D3
C9	MS Tower	Char	4.25	$\mu\text{g/g}$	D2
C10	MS Tower	Char	9.44	$\mu\text{g/g}$	D2
C11	MS Tower	Char	4.55	$\mu\text{g/g}$	D2
C12	MS Tower	Char	9.12	$\mu\text{g/g}$	D2
C13	MS Tower	Char	5.07	$\mu\text{g/g}$	D2
C14	MS Tower	Char	10.8	$\mu\text{g/g}$	D2
C16	MS Tower	Char	7.95	$\mu\text{g/g}$	D2
C18	MS Tower	Char	--	$\mu\text{g/g}$	D2, <LOD
C4	QX Tower	Char	20.1	$\mu\text{g/g}$	D2
C5	QX Tower	Char	41.7	$\mu\text{g/g}$	D2
C6	QX Tower	Char	122	$\mu\text{g/g}$	D2
C7	QX Tower	Char	71.5	$\mu\text{g/g}$	D2



PFCA	Sample ID	Matrix	Conc.	Unit	Flag(s)*
C8	QX Tower	Char	1430	µg/g	D3
C9	QX Tower	Char	36.2	µg/g	D2
C10	QX Tower	Char	74.8	µg/g	D2
C11	QX Tower	Char	52.2	µg/g	D2
C12	QX Tower	Char	87.5	µg/g	D2
C13	QX Tower	Char	62.9	µg/g	D2
C14	QX Tower	Char	79.3	µg/g	D2
C16	QX Tower	Char	28.4	µg/g	D2, <LOQ
C18	QX Tower	Char	3.14	µg/g	D2, <LOQ
C4	MA Tower	Char	--	µg/g	D2, <LOD
C5	MA Tower	Char	--	µg/g	D2, <LOD
C6	MA Tower	Char	0.212	µg/g	D2, <LOQ
C7	MA Tower	Char	--	µg/g	D2, <LOD
C8	MA Tower	Char	2.55	µg/g	D2, <LOQ
C9	MA Tower	Char	--	µg/g	D2, <LOD
C10	MA Tower	Char	--	µg/g	D2, <LOD
C11	MA Tower	Char	--	µg/g	D2, <LOD
C12	MA Tower	Char	0.100	µg/g	D2, <LOQ
C13	MA Tower	Char	0.095	µg/g	D2, <LOQ
C14	MA Tower	Char	0.193	µg/g	D2
C16	MA Tower	Char	0.423	µg/g	D2
C18	MA Tower	Char	0.357	µg/g	D2, <LOQ
C4	EPAORDS1	Soil	194	pg/g	UD
C5	EPAORDS1	Soil	389	pg/g	UD, <LOQ
C6	EPAORDS1	Soil	1270	pg/g	UD
C7	EPAORDS1	Soil	615	pg/g	UD
C8	EPAORDS1	Soil	7420	pg/g	D1, <LOQ
C9	EPAORDS1	Soil	240	pg/g	UD
C10	EPAORDS1	Soil	238	pg/g	UD, <LOQ
C11	EPAORDS1	Soil	90.0	pg/g	UD, <LOQ
C12	EPAORDS1	Soil	--	pg/g	UD, <LOD
C13	EPAORDS1	Soil	--	pg/g	UD, <LOD
C14	EPAORDS1	Soil	--	pg/g	UD, <LOD
C16	EPAORDS1	Soil	--	pg/g	UD, <LOD
C18	EPAORDS1	Soil	--	pg/g	UD, <LOD
C4	EPAORDS2	Soil	--	pg/g	UD, <LOD
C5	EPAORDS2	Soil	--	pg/g	UD, <LOD
C6	EPAORDS2	Soil	175	pg/g	UD, <LOQ
C7	EPAORDS2	Soil	540	pg/g	UD
C8	EPAORDS2	Soil	6950	pg/g	UD
C9	EPAORDS2	Soil	--	pg/g	UD, <LOD
C10	EPAORDS2	Soil	34.3	pg/g	UD, <LOQ

PFCA	Sample ID	Matrix	Conc.	Unit	Flag(s)*
C11	EPAORDS2	Soil	--	pg/g	UD, <LOD
C12	EPAORDS2	Soil	--	pg/g	UD, <LOD
C13	EPAORDS2	Soil	--	pg/g	UD, <LOD
C14	EPAORDS2	Soil	--	pg/g	UD, <LOD
C16	EPAORDS2	Soil	--	pg/g	UD, <LOD
C18	EPAORDS2	Soil	--	pg/g	UD, <LOD
C4	EPAORDS3	Soil	--	pg/g	UD, <LOD
C5	EPAORDS3	Soil	18.9	pg/g	UD, <LOQ
C6	EPAORDS3	Soil	56.1	pg/g	UD, <LOQ
C7	EPAORDS3	Soil	165	pg/g	UD, <LOQ
C8	EPAORDS3	Soil	1140	pg/g	UD
C9	EPAORDS3	Soil	21.4	pg/g	UD, <LOQ
C10	EPAORDS3	Soil	19.4	pg/g	UD, <LOQ
C11	EPAORDS3	Soil	5.70	pg/g	UD, <LOQ
C12	EPAORDS3	Soil	--	pg/g	UD, <LOD
C13	EPAORDS3	Soil	--	pg/g	UD, <LOD
C14	EPAORDS3	Soil	--	pg/g	UD, <LOD
C16	EPAORDS3	Soil	--	pg/g	UD, <LOD
C18	EPAORDS3	Soil	--	pg/g	UD, <LOD

\* Flags defined:

UD = undiluted

D1 = 10-fold dilution

D2 = 10<sup>4</sup>-fold dilution

D3 = 10<sup>5</sup>-fold dilution

<LOQ = Less than limit of quantitation (defined as exceeding process blanks  $p > 0.05$  but  $\leq 0.001$ )

<LOD = Less than limit of detection (defined by  $p \leq 0.05$ )

Appendix A-7  
ORD Report #2  
July 24, 2018



**UNITED STATES ENVIRONMENTAL PROTECTION AGENCY**  
NATIONAL EXPOSURE RESEARCH LABORATORY  
OFFICE OF RESEARCH AND DEVELOPMENT  
RESEARCH TRIANGLE PARK, NC 27711

July 24, 2018

Mr. Clark Freise, Assistant Commissioner  
New Hampshire Department of Environmental Services  
29 Hazen Drive  
PO Box 95  
Concord, New Hampshire 03301

Dr. Mr. Freise,

I am pleased to provide you with the attached report which presents additional results of analyses of per- and polyfluoroalkyl substances (PFAS) concentrations in char and soil samples collected by the New Hampshire Department of Environmental Services (NH DES) staff. This report provides results of non-targeted analyses of the same three soil and three char samples that were previously analyzed for C4-C18 "legacy" perfluorocarboxylates using targeted methods and delivered to the NH DES in our April 4, 2018 report. The current report is significant in identifying the presence of two PFAS series that to our knowledge has not been previously reported as an environmental contaminant.

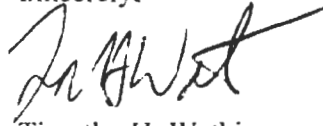
It is our understanding that this information was requested by the NH DES to help in their ongoing investigation into the presence of PFAS in the environment near manufacturing facilities of interest. This request relates to our research capabilities and interests applying targeted and non-targeted analysis methods for discovery of the nature and extent of PFAS environmental occurrence.

The U.S. Environmental Protection Agency continues to develop analytical methods for many PFAS compounds in various media including some of those included in this report. The data enclosed provides information related to the concentration of certain PFAS in the media sampled. In this report we do not interpret exposure or risk from these values. The EPA does not currently have health based standards, toxicity factors or associated risk levels for PFAS, other than perfluorooctanoic acid (PFOA), perfluorooctane sulfonate (PFOS), and perfluorobutanesulfonic acid (PFBS). Therefore, while the data presented indicate the presence of two novel PFAS series, no conclusions can be made related to human or environmental exposure and risk.

Thank you for inviting us to be part of this effort that helps to further both the EPA's and New Hampshire's understanding of an important issue in the state. Providing this type of support to the NH DES aligns well with our research capabilities and interests in applying targeted and non-targeted analysis methods. It also demonstrates our commitment to, and the relevance of our research in support of, cooperative federalism to address pressing environmental health concerns of New Hampshire residents.

If you have any questions or concerns, do not hesitate to contact me at (919) 541-2106 or via email at [watkins.tim@epa.gov](mailto:watkins.tim@epa.gov) or Tim Buckley at (919) 541-2454 or via email at [buckley.timothy@epa.gov](mailto:buckley.timothy@epa.gov). I look forward to our continued work together.

Sincerely,

A handwritten signature in black ink, appearing to read 'Tim H. Watkins', with a long horizontal stroke extending to the right.

Timothy H. Watkins  
Director

Attachment

cc: Meghan Cassidy, USEPA, Region 1  
Deb Szaro, USEPA, Region 1  
Jeff Morris, USEPA OPPT  
Betsy Behl, USEPA, OW  
Peter Grevatt, USEPA, OW  
Andy Gillespie, USEPA, ORD  
Timothy Buckley, USEPA, ORD

## **Technical Report #2: ORD Technical Support to New Hampshire: Non-Targeted PFAS Measurements in Char and Soil**

**Date:** July 24, 2018

### **Report Team**

- **Laboratory Chemists:** Drs. John Washington, Mark Strynar, Andy Lindstrom, Seth Newton, Thomas Jenkins, and James McCord
- **Quality Assurance Review:** Sania Tong-Argao and Brittany Stuart
- **Management Coordination and Review:** Drs. Myriam Medina-Vera, Jack Jones, Adam Biales, and Brian Schumacher
- **Report Preparation:** Dr. Tim Buckley

### **Summary of Methods and Results**

New Hampshire Department of Environmental Services (NH DES), in coordination with Region 1, requested ORD's technical support in analyzing PFAS in environmental samples potentially impacted by industrial sites within the state. NH DES assumed responsibility for the collection of samples and their shipment to our laboratories. ORD was responsible for sample extraction and analysis. We are providing the results of our analysis as they become available. Our first report dated April 4, 2018 provided targeted analysis results for C4-C18 "legacy" perfluorocarboxylates in 3 soil and 3 char samples.

This is our second report and it includes non-targeted analysis results conducted by Dr. John Washington of the same soil and char samples previously reported (Report No. 1). The non-targeted analysis differs from the targeted in that chemical identification and quantification does not have the benefit of being based on a known standard. Accordingly, there is more uncertainty both in terms of identification and concentration estimation.

The PFAS reported here were identified and quantified using non-targeted analysis methods described within an approved Quality Assurance Project Plan (QAPP)<sup>1</sup>. These methods are also generally described in Rankin et al., 2015.<sup>2</sup> In brief, each sample was divided into three ~1 g aliquots and extracted and analyzed in triplicate. Extracts were analyzed by liquid chromatography / mass spectrometry using a Waters Acquity UPLC coupled to a Waters Xevo G2-XS QTOF for non-targeted identification followed by a Waters Acquity UPLC coupled to a Waters Quattro Premier XE tandem for quantitation. The non-targeted PFAS are identified based on a combination of high-resolution mass spectral data along with patterns of fragmentation. Without the benefit of a standard, we quantify based on the fully fluorinated homologue for which we do have a standard. In effect, our quantification of the non-targeted analyte, in this

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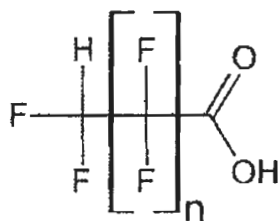
<sup>1</sup> Strynar, M.; Washington, J.; Lindstrom, A.; Henderson, W. 2017. Quality Assurance Project Plan: Non-Targeted Analyses of Per- and Polyfluoroalkyl Substances (PFAS) for New Hampshire Department of Environmental Services (NHDES). D-EMMD-PHCB-015-QAPP-01.

<sup>2</sup> K. Rankin, S. A. Mabury, T. M. Jenkins, J. W. Washington, A North American and global survey of perfluoroalkyl substances in surface Soil: Distribution patterns and mode of occurrence. *Chemosphere* 161, 333-341 (2015).

case the hydrogenated carboxylic acid, assumes that the mass spectrometer responds to the fully fluorinated species as it does the hydrogenated, i.e. yielding identical chromatographic peak areas. A calibration curve is used for quantification, however, the curve is developed for chemicals for which we have a standard and that are similar to our non-targeted analyte. In most cases, the standard is the fully fluorinated version of the PFAS we are quantifying as noted in Table 3. Our experience with these chemicals has shown that this means of estimation often is within an order of magnitude uncertainty. Some of the sample extracts required dilution so that concentrations were within the acceptable range of the calibration curve. The reported results have been adjusted for each dilution factor and flagged accordingly. These analyses were performed on samples and process blanks using internal-calibration curves for quantitation. The mean value of the triplicate analysis is reported.

Measurement precision was estimated by the relative standard deviation (RSD) from triplicate analysis of each sample. For char samples, the median RSD across all of the compounds was 4.4% and ranged from 0.7% to 31%. We observed greater variability in soil where concentrations were much lower. The median RSD for soil was 66% and ranged from 8.4% to 170%. Field blanks were not provided so limits of detection (LOD) and quantification (LOQ) were determined from process blanks at  $p \leq 0.05$  and  $p \leq 0.001$ , respectively. Reported concentrations were corrected for any low detections in process blanks.

The non-targeted analysis identified two PFAS series not previously reported to our knowledge as an environmental contaminant. The first is a PFAS carboxylic acid series that ranges from C6 to C20 where there is a single hydrogen substitution for fluorine. Our identification of the chemicals in this series is provided in Table 1. We are confident in chemical identities based on mass-spectral data including high resolution mass and fragmentation data. However, at present we cannot determine the exact location of the hydrogen substitution, and therefore we have not specified a CAS number. The generic hydrogenated polyfluorinated carboxylic acid (HPFCA) structure is given in Figure 1 with the hydrogen arbitrarily placed in the terminal position.

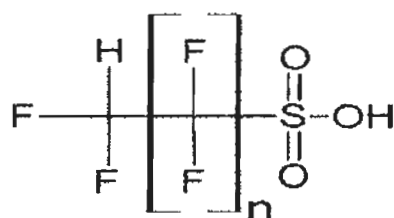


**Figure 1.** Generic structure of hydrogenated polyfluorinated carboxylic acid (HPFCA). At present, the exact position of the hydrogen is undetermined.

**Table 1. PFAS Single Hydrogen Substituted Carboxylic Acid Series Identified Using Non-Targeted Analyses that are the Subject of this Report.**

Carbon No.	Compound Name	Acronym	Anion Formula
HC6	Hydro-polyfluorohexanoic acid	HPFH <sub>x</sub> A	HC <sub>6</sub> F <sub>10</sub> O <sub>2</sub>
HC7	Hydro-polyfluoroheptanoic acid	HPFH <sub>p</sub> A	HC <sub>7</sub> F <sub>12</sub> O <sub>2</sub>
HC8	Hydro-polyfluorooctanoic acid	HPFOA	HC <sub>8</sub> F <sub>14</sub> O <sub>2</sub>
HC9	Hydro-polyfluorononanoic acid	HPFNA	HC <sub>9</sub> F <sub>16</sub> O <sub>2</sub>
HC10	Hydro-polyfluorodecanoic acid	HPFDA	HC <sub>10</sub> F <sub>18</sub> O <sub>2</sub>
HC11	Hydro-polyfluoroundecanoic acid	HPFUA	HC <sub>11</sub> F <sub>20</sub> O <sub>2</sub>
HC12	Hydro-polyfluorododecanoic acid	HPFDoA	HC <sub>12</sub> F <sub>22</sub> O <sub>2</sub>
HC13	Hydro-polyfluorotridecanoic acid	HPFTrA	HC <sub>13</sub> F <sub>24</sub> O <sub>2</sub>
HC14	Hydro-polyfluorotetradecanoic acid	HPFTeA	HC <sub>14</sub> F <sub>26</sub> O <sub>2</sub>
HC15	Hydro-polyfluoropentadecanoic acid	HPFPDA	HC <sub>15</sub> F <sub>28</sub> O <sub>2</sub>
HC16	Hydro-polyfluorohexadecanoic acid	HPFH <sub>x</sub> DA	HC <sub>16</sub> F <sub>30</sub> O <sub>2</sub>
HC17	Hydro-polyfluoroheptadecanoic acid	HPFH <sub>p</sub> DA	HC <sub>17</sub> F <sub>32</sub> O <sub>2</sub>
HC18	Hydro-polyfluorooctadecanoic acid	HPFODA	HC <sub>18</sub> F <sub>34</sub> O <sub>2</sub>
HC19	Hydro-polyfluorononadecanoic acid	HPFNDA	HC <sub>19</sub> F <sub>36</sub> O <sub>2</sub>
HC20	Hydro-polyfluoroicosanoic acid	HPFLA	HC <sub>20</sub> F <sub>38</sub> O <sub>2</sub>

The second series discovered is a polyfluorinated sulfonic acid series, again with a single hydrogen substitution (Table 2). The generic hydrogenated polyfluorinated sulfonic acid (HPFSA) structure is given in Figure 2 with the hydrogen arbitrarily placed in the terminal position.



**Figure 2.** The generic structure of hydrogenated polyfluorinated sulfonic acid (HPFSA). At present, the exact position of the hydrogen is undetermined.



**Table 2. PFAS Single Hydrogen Substituted Sulfonic Acid Series Identified Using Non-Targeted Analyses that are the Subject of this Report.**

<b>Carbon No.</b>	<b>Compound Name</b>	<b>Acronym</b>	<b>Anion Formula</b>
HSC4	Hydro-polyfluorobutanesulfonate	HPFBS	HC <sub>4</sub> F <sub>8</sub> SO <sub>3</sub>
HSC5	Hydro-polyfluoropentanesulfonate	HPFPS	HC <sub>5</sub> F <sub>10</sub> SO <sub>3</sub>
HSC6	Hydro-polyfluorohexanesulfonate	HPFHxS	HC <sub>6</sub> F <sub>12</sub> SO <sub>3</sub>
HSC7	Hydro-polyfluoroheptanesulfonate	HPFHpS	HC <sub>7</sub> F <sub>14</sub> SO <sub>3</sub>
HSC8	Hydro-polyfluorooctanesulfonate	HPFOS	HC <sub>8</sub> F <sub>16</sub> SO <sub>3</sub>
HSC9	Hydro-polyfluorononanesulfonate	HPFNS	HC <sub>9</sub> F <sub>18</sub> SO <sub>3</sub>
HSC10	Hydro-polyfluorodecanesulfonate	HPFDS	HC <sub>10</sub> F <sub>20</sub> SO <sub>3</sub>
HSC11	Hydro-polyfluoroundecanesulfonate	HPFUS	HC <sub>11</sub> F <sub>22</sub> SO <sub>3</sub>
HSC12	Hydro-polyfluorododecanesulfonate	HPFDoS	HC <sub>12</sub> F <sub>24</sub> SO <sub>3</sub>
HSC13	Hydro-polyfluorotridecanesulfonate	HPFTrS	HC <sub>13</sub> F <sub>26</sub> SO <sub>3</sub>
HSC14	Hydro-polyfluorotetradecanesulfonate	HPFTeS	HC <sub>14</sub> F <sub>28</sub> SO <sub>3</sub>
HSC15	Hydro-polyfluoropentadecanesulfonate	HPFPDS	HC <sub>15</sub> F <sub>30</sub> SO <sub>3</sub>
HSC16	Hydro-polyfluorohexadecanesulfonate	HPFHxDS	HC <sub>16</sub> F <sub>32</sub> SO <sub>3</sub>
HSC17	Hydro-polyfluoroheptadecanesulfonate	HPFHpDS	HC <sub>17</sub> F <sub>34</sub> SO <sub>3</sub>
HSC18	Hydro-polyfluorooctadecanesulfonate	HPFODS	HC <sub>18</sub> F <sub>36</sub> SO <sub>3</sub>

As was observed for perfluorocarboxylates in our first report, the hydrogen substituted analogues tended to occur at higher concentrations in char relative to soil samples. The stack char concentrations of HPFCA ranged from <LOD (limit of detection) to 140 µg/g (HC10). (Note, the upper range is uncertain because some values are only specified as exceeding the calibration range.) Among the char samples, “NHCharMA” levels generally exceeding levels observed for char from “MS” or “QX” stack samples.

Soil concentrations were orders of magnitude lower than char samples. As a result, we report soil levels in pg/g instead of µg/g. The observed variation ranges from <LOD to 157 pg/g. As was observed for PFCA in our first report, concentrations of the hydrogenated substituted PFCA tended to be much higher in soil sample “S1” relative to “S2” or “S3.” Having identified these novel PFAS in char and soil, we will also be looking for these same novel PFAS in water and air samples to be included in later reports.

At this time we provide concentration estimates for only HPFCA because we observed higher concentrations of the HPFCA series relative to HPFSA. Based on a comparison of peak areas between the carbon equivalents in the two series, HPFCA levels in char exceeded HPFSA by a factor of 2 to 50 except for C6 and C8 where HPFSA was greater by a factor of 10 and 1.2, respectively (results not provided). We would not expect to find HPFSA in soil at appreciable

levels because char PFAS concentrations generally exceed soil, and levels of HPFSA were generally low in char. As mentioned above, the concentrations reported below in Table 3 are considered semi-quantitative, likely within an order of magnitude of the actual value.

**Table 3. Concentration Estimates of Single Hydrogen Substituted PFAS Carboxylic Acids from Non-Targeted Analysis.**

Carbon No.*	Sample ID	Matrix	Conc.	Unit	Flag(s)**
HC6	NHCharMS	Char	2.98	µg/g	D1
HC7	NHCharMS	Char	0.631	µg/g	D1
HC8	NHCharMS	Char	4.79	µg/g	D1
HC9	NHCharMS	Char	10.6	µg/g	D1
HC10	NHCharMS	Char	9.32	µg/g	D1
HC11	NHCharMS	Char	2.64	µg/g	D1
HC12	NHCharMS	Char	7.95	µg/g	D1
HC13	NHCharMS	Char	2.99	µg/g	D1
HC14	NHCharMS	Char	10.8	µg/g	D1
HC15 as C14	NHCharMS	Char	5.75	µg/g	D1
HC16	NHCharMS	Char	16.1	µg/g	D1
HC17 as C16	NHCharMS	Char	4.02	µg/g	D1
HC18	NHCharMS	Char	6.30	µg/g	D1
HC19 as C18	NHCharMS	Char	1.24	µg/g	D1, <LOQ
HC20 as C20	NHCharMS	Char	0.392	µg/g	D1, <LOQ
HC6	NHCharQX	Char	--	µg/g	D1, <LOD
HC7	NHCharQX	Char	0.142	µg/g	D1, <LOQ
HC8	NHCharQX	Char	--	µg/g	D1, <LOD
HC9	NHCharQX	Char	1.71	µg/g	D1, <LOQ
HC10	NHCharQX	Char	--	µg/g	D1, <LOD
HC11	NHCharQX	Char	0.620	µg/g	D1
HC12	NHCharQX	Char	--	µg/g	D1, <LOD
HC13	NHCharQX	Char	0.547	µg/g	D1, <LOQ
HC14	NHCharQX	Char	0.592	µg/g	D1, <LOQ
HC15 as C14	NHCharQX	Char	0.885	µg/g	D1
HC16	NHCharQX	Char	0.379	µg/g	D1, <LOQ
HC17 as C16	NHCharQX	Char	0.220	µg/g	D1, <LOQ
HC18	NHCharQX	Char	0.213	µg/g	D1, E1
HC19 as C18	NHCharQX	Char	--	µg/g	D1, <LOD
HC20 as C18	NHCharQX	Char	--	µg/g	D1, <LOD
HC6	NHCharMA	Char	36.4	µg/g	D1
HC7	NHCharMA	Char	8.66	µg/g	D1
HC8	NHCharMA	Char	67.7	µg/g	D1
HC9	NHCharMA	Char	101	µg/g	D1
HC10	NHCharMA	Char	140	µg/g	D1
HC11	NHCharMA	Char	26.6	µg/g	D1
HC12	NHCharMA	Char	> 43.3	µg/g	D1, E2
HC13	NHCharMA	Char	22.5	µg/g	D1
HC14	NHCharMA	Char	> 43.3	µg/g	D1, E2
HC15	NHCharMA	Char	42.9	µg/g	D1

**Table 3. Concentration Estimates of Single Hydrogen Substituted PFAS Carboxylic Acids from Non-Targeted Analysis.**

Carbon No.*	Sample ID	Matrix	Conc.	Unit	Flag(s)**
HC16	NHCharMA	Char	> 21.8	µg/g	D1, E2
HC17	NHCharMA	Char	31.7	µg/g	D1
HC18	NHCharMA	Char	> 21.8	µg/g	D1, E2
HC19	NHCharMA	Char	1.37	µg/g	D1
HC20	NHCharMA	Char	0.356	µg/g	D1, <LOQ
HC6	NHEPAORD-S1	Soil	50.9	pg/g	UD, <LOQ
HC7	NHEPAORD-S1	Soil	3.11	pg/g	UD, <LOQ
HC8	NHEPAORD-S1	Soil	50.9	pg/g	UD
HC9	NHEPAORD-S1	Soil	113	pg/g	UD, <LOQ
HC10	NHEPAORD-S1	Soil	122	pg/g	UD
HC11	NHEPAORD-S1	Soil	28.3	pg/g	UD
HC12	NHEPAORD-S1	Soil	104	pg/g	UD
HC13	NHEPAORD-S1	Soil	23.8	pg/g	UD
HC14	NHEPAORD-S1	Soil	157	pg/g	UD
HC15 as C14	NHEPAORD-S1	Soil	33.9	pg/g	UD
HC16	NHEPAORD-S1	Soil	142	pg/g	UD
HC17 as C16	NHEPAORD-S1	Soil	8.95	pg/g	UD, <LOQ
HC18	NHEPAORD-S1	Soil	13.6	pg/g	UD, <LOQ
HC19 as C18	NHEPAORD-S1	Soil	--	pg/g	UD, <LOD
HC20 as C18	NHEPAORD-S1	Soil	--	pg/g	UD, <LOD
HC6	NHEPAORD-S2	Soil	--	pg/g	UD, <LOD
HC7	NHEPAORD-S2	Soil	--	pg/g	UD, <LOD
HC8	NHEPAORD-S2	Soil	--	pg/g	UD, <LOD
HC9	NHEPAORD-S2	Soil	--	pg/g	UD, <LOD
HC10	NHEPAORD-S2	Soil	--	pg/g	UD, <LOD
HC11	NHEPAORD-S2	Soil	--	pg/g	UD, <LOD
HC12	NHEPAORD-S2	Soil	--	pg/g	UD, <LOD
HC13	NHEPAORD-S2	Soil	--	pg/g	UD, <LOD
HC14	NHEPAORD-S2	Soil	--	pg/g	UD, <LOD
HC15 as C14	NHEPAORD-S2	Soil	--	pg/g	UD, <LOD
HC16	NHEPAORD-S2	Soil	--	pg/g	UD, <LOD
HC17 as C16	NHEPAORD-S2	Soil	--	pg/g	UD, <LOD
HC18	NHEPAORD-S2	Soil	--	pg/g	UD, <LOD
HC19 as C18	NHEPAORD-S2	Soil	--	pg/g	UD, <LOD
HC20 as C18	NHEPAORD-S2	Soil	--	pg/g	UD, <LOD
HC6	NHEPAORD-S3	Soil	--	pg/g	UD, <LOD
HC7	NHEPAORD-S3	Soil	--	pg/g	UD, <LOD
HC8	NHEPAORD-S3	Soil	--	pg/g	UD, <LOD
HC9	NHEPAORD-S3	Soil	14.3	pg/g	UD, <LOQ
HC10	NHEPAORD-S3	Soil	--	pg/g	UD, <LOD
HC11	NHEPAORD-S3	Soil	--	pg/g	UD, <LOD
HC12	NHEPAORD-S3	Soil	--	pg/g	UD, <LOD
HC13	NHEPAORD-S3	Soil	2.93	pg/g	UD, <LOQ
HC14	NHEPAORD-S3	Soil	11.2	pg/g	UD, <LOQ
HC15 as C14	NHEPAORD-S3	Soil	--	pg/g	UD, <LOD

**Table 3. Concentration Estimates of Single Hydrogen Substituted PFAS Carboxylic Acids from Non-Targeted Analysis.**

Carbon No.*	Sample ID	Matrix	Conc.	Unit	Flag(s)**
HC16	NHEPAORD-S3	Soil	10.9	pg/g	UD, <LOQ
HC17 as C16	NHEPAORD-S3	Soil	--	pg/g	UD, <LOD
HC18	NHEPAORD-S3	Soil	--	pg/g	UD, <LOD
HC19 as C18	NHEPAORD-S3	Soil	--	pg/g	UD, <LOD
HC20 as C18	NHEPAORD-S3	Soil	--	pg/g	UD, <LOD

\*Unless specified otherwise, the hydrogen substituted PFAS was quantified based on the fully fluorinated analogue.

\*\* Flags defined:

- UD = undiluted
- D1 = 10-fold dilution
- <LOQ = values are less than LOQ but exceed Limit of Detection (LOD) defined as exceeding process blanks at  $P < 0.05$  level of significance
- <LOD = Less than limit of detection and not significantly different than process blanks
- E1= sample/analyte exceeded established precision criteria of  $\pm 30\%$
- E2= the calibration range for the corresponding PFCA was exceeded so that the reported values are greater than the highest calibration standard. The calibration range varied by carbon length.

Appendix A-8  
ORD Report #3  
October 4, 2018



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
NATIONAL EXPOSURE RESEARCH LABORATORY  
RESEARCH TRIANGLE PARK, NC 27711

OFFICE OF  
RESEARCH AND DEVELOPMENT

October 4, 2018

Mr. Clark Freise, Assistant Commissioner  
New Hampshire Department of Environmental Services (NHDES)  
29 Hazen Drive  
P.O. Box 95  
Concord, New Hampshire 03301

Dear Mr. Freise:

I am pleased to provide the attached 3<sup>rd</sup> and 4<sup>th</sup> report from our ongoing collaborative technical support to NHDES assisting with concern over PFAS environmental contamination associated with manufacturing sites. These reports are in response to your request of June 22, 2017 asking for laboratory assistance analyzing per- and polyfluoroalkyl substances (PFAS) in environmental samples. The enclosed Report #3 provides results for surface and ground water samples. Report #4 provides the results of stack sampling.

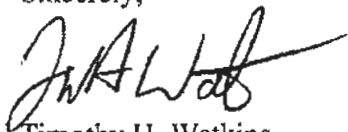
It is our understanding that this information was requested by NHDES to help in your ongoing investigation into the presence of per- and polyfluorinated alkyl substances (PFAS) in the environment near manufacturing facilities of interest. This request relates to our research capabilities and interests applying targeted and non-targeted analysis methods for discovery of the nature and extent of PFAS environmental occurrence that may be potentially associated with industrial releases. EPA continues to develop analytical methods for many PFAS compounds in various media including some of those included in this report. We are providing the results of our analysis as they become available.

In this report, we do not interpret exposure or risk from these values. EPA does not currently have health-based standards, toxicity factors, or associated risk levels for PFAS, other than perfluorooctanoic acid (PFOA), perfluorocatane sulfonate (PFOS), and perfluorobutanesulfonic acid (PFBS). While the data provided in the attached reports indicate the presence of PFAS in water samples, no conclusions can be made related to human or environmental exposure and risk.

Thank you for inviting us to be part of this effort that helps to further both EPA's and New Hampshire's understanding of an important issue in the state. This is just one of many Agency efforts that demonstrates EPA's commitment to cooperative federalism.

If you have any questions or concerns, do not hesitate to contact me at (919) 541-2107 or via email at [watkins.tim@epa.gov](mailto:watkins.tim@epa.gov) or Tim Buckley at (919) 541-2454 or via email at [buckley.timothy@epa.gov](mailto:buckley.timothy@epa.gov). I look forward to our continued work together.

Sincerely,

A handwritten signature in black ink, appearing to read 'Tim H. Watkins', with a stylized flourish at the end.

Timothy H. Watkins

Director

National Exposure Research Laboratory

Office of Research and Development

Enclosure

CC: Meghan Cassidy, USEPA, Region 1  
Deb Szaro, USEPA, Region 1  
Jeff Morris, USEPA OPPT  
Betsy Behl, USEPA, OW  
Peter Grevatt, USEPA, OW  
Andy Gillespie, USEPA, ORD  
Timothy Buckley, USEPA, ORD  
Cindy Sonich-Mullen, USEPA, ORD

### ORD Report #3: Technical Support to New Hampshire - Targeted PFAS Measurements in Water

**Date:** October 4, 2018

New Hampshire Department of Environmental Services (NHDES), in coordination with EPA Region 1, requested technical support from EPA's Office of Research and Development (ORD) in analyzing PFAS in environmental samples potentially impacted by industrial sites within the state. NHDES assumed responsibility for the collection of samples and their shipment to our laboratories. ORD was responsible for sample extraction and analysis of PFAS. ORD's analysis and support team for this report are listed in Table 1.

**Table 1. EPA Office of Research and Development analysis and report team.**

Responsibility	Personnel
Laboratory chemistry	Mark Strynar (team lead), James McCord, Seth Newton
Quality assurance review	Andy Lindstrom, Sania Tong-Argao
Management coordination and review	Myriam Medina-Vera, Brian Schumacher, Timothy Buckley
Report preparation	Kate Sullivan

The current report includes results for water samples collected by NHDES on September 27, 2017 (n=25) in containers provided by ORD. Samples were sent to and analyzed under the direction of Dr. Mark Strynar at ORD's laboratories in Research Triangle Park, NC. ORD laboratory personnel were blind to sampling location. The PFAS analytes targeted for analysis are given in Table 2. These analytes were selected because previous NHDES reports have shown them to be of concern.

Water samples were analyzed by Liquid Chromatography Mass Spectrometry (LC-MS) according to methods described within an approved Quality Assurance Project Plan (QAPP)<sup>1</sup>. PFAS concentrations were determined against a standard calibration curve derived from authentic standards using a traditional targeted analysis approach.

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<sup>1</sup> National Exposure Research Laboratory, Quality Assurance Project Plan: Non-Targeted Analyses of Per- and Polyfluoroalkyl Substances (PFAS) for New Hampshire Department of Environmental Services (NHDES), October 2, 2017.



**Table 2. PFAS Analytes Measured**

Short Name	Chemical Name	Formula	CAS no.
GenX	Perfluoro(2-methyl-3-oxahexanoic) acid	C <sub>6</sub> HF <sub>11</sub> O <sub>3</sub>	13252-13-6
PFBA	Perfluorobutanoic Acid	C <sub>4</sub> HF <sub>7</sub> O <sub>2</sub>	375-22-4
PFPeA	Perfluoropentanoic Acid	C <sub>5</sub> HF <sub>9</sub> O <sub>2</sub>	2706-90-3
PFHxA	Perfluorohexanoic Acid	C <sub>6</sub> HF <sub>11</sub> O <sub>2</sub>	307-24-4
PFHpA	Perfluoroheptanoic Acid	C <sub>7</sub> HF <sub>13</sub> O <sub>2</sub>	375-85-9
PFOA	Perfluorooctanoic Acid	C <sub>8</sub> HF <sub>15</sub> O <sub>2</sub>	335-67-1
PFNA	Perfluorononanoic Acid	C <sub>9</sub> HF <sub>17</sub> O <sub>2</sub>	375-95-1
PFDA	Perfluorodecanoic Acid	C <sub>10</sub> HF <sub>19</sub> O <sub>2</sub>	335-76-2
PFBS	Perfluorobutane Sulfonate	C <sub>4</sub> HF <sub>9</sub> SO <sub>3</sub>	375-73-5
PFHxS	Perfluorohexane Sulfonate	C <sub>6</sub> HF <sub>13</sub> SO <sub>3</sub>	355-46-4
PFOS	Perfluorooctane Sulfonate	C <sub>8</sub> HF <sub>17</sub> SO <sub>3</sub>	1763-23-1

## Results

Quality control results indicated analyses were within expected performance specifications. For GenX, we observed our standards to be within  $\pm 22\%$  of our calibration curve over the range of 10 to 1000 ng/L. Quality Control spikes (100 and 500 ng/L) were within 12% of the target concentration. GenX was not detected in any field or laboratory blanks.

For the remaining PFAS analytes, we observed deviations from the calibration curve for lower range standards (i.e. 10, 50, and 100 ng/L) by as much as 74%, 52%, and 51.3%, respectively. For the 25 ng/L and higher standards, concentrations were within  $\pm 21.8\%$  of the calibration curve. QC spike samples (100 and 500 ng/L) were within 32% of the target except for PDFA, which deviated by 62.6%.

Concentration results for the 25 water samples are presented in Table 3. Summary findings include:

- GenX was detected in one sample. All other samples were below the detection limit.
- Concentrations of other PFAS varied by sample and analyte. The range of concentrations for other analytes varied from less than the limit of detection to 2,200 ng/L.
- Most of the samples had measurable concentrations of one or more of the legacy PFAS, and many had measurable concentrations of multiple analytes.
- PFOA was consistently present at the highest concentration across the 25 samples. PFOS and PFBS were observed in multiple samples.

**Table 3. PFAS Concentrations determined with targeted analysis in ng/L.**

Sample ID	GenX	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFBS	PFHxS	PFOS	Sum
EPAORD 001	<LOQ	11.1	19.7	31.7	49.2	227	30.1	48.2	9.92	23.9	36.4	487
EPAORD 002	<LOQ	-	-	-	-	-	-	10.8	-	-	-	10.8
EPAORD 003	<LOQ	-	-	-	-	-	-	45.0	-	-	19.6	64.6
EPAORD 004	<LOQ	35.9	153	168	124	580	-	39.7	12.5	21.1	37.9	1,170
EPAORD 005	<LOQ	36.5	143	155	126	578	-	11.3	18.5	15.0	37.6	1,120
EPAORD 006	<LOQ	160	577	713	679	2,270	23.3	12.6	9.79	38.2	278	4,760
EPAORD 007	<LOQ	-	9.82	18.5	25.8	101	-	-	-	-	-	155
EPAORD 008	<LOQ	-	-	-	14.6	70.3	-	-	-	-	-	84.9
EPAORD 009	<LOQ	35.0	74.8	95.3	71.0	397	-	-	-	-	-	673
EPAORD 010	<LOQ	21.1	73.9	109	134	497	-	-	13.6	12.3	-	861
EPAORD 011	<LOQ	73.8	286	381	500	1,460	-	-	21.7	21.7	-	2,750
EPAORD 012	<LOQ	10.9	30.4	50.1	92.6	452	-	-	9.55	86.4	-	732
EPAORD 013	<LOQ	9.88	31.9	49.8	116	448	-	-	-	88.7	-	744
EPAORD 014	<LOQ	-	-	-	-	-	-	-	-	-	-	0.0
EPAORD 015	<LOQ	-	23.0	17.4	18.8	100	-	-	11.5	-	-	171
EPAORD 016	35.4	-	18.7	26.6	35.5	117	-	-	14.2	-	-	212
EPAORD 017	<LOQ	-	-	15.6	28.5	180	-	-	34.8	9.93	-	269
EPAORD 018	<LOQ	-	-	-	-	61.7	-	-	-	-	-	61.7
EPAORD 019	<LOQ	-	11.8	15.5	19.8	96.1	-	-	57.5	-	-	201
EPAORD 020	<LOQ	-	-	-	10.6	61.2	-	-	-	9.64	-	81.5
EPAORD 021	<LOQ	-	-	-	10.7	61.7	-	-	-	-	-	72.4
EPAORD 022	<LOQ	-	18.9	30.5	51.1	166	-	-	15.3	17.4	-	299
EPAORD 023	<LOQ	9.72	56.4	69.7	100	521	-	-	-	19.1	-	776
EPAORD 024	<LOQ	-	-	-	13.5	34.7	-	-	-	-	-	48.3
EPAORD 901	<LOQ	-	-	18.1	53.7	419	-	-	-	17.5	-	508

&lt;LOQ = Less than limit of quantitation for GenX is 10 ng/L - (dash) = Analyte not detected

Appendix A-9  
ORD Report #4  
October 4, 2018



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
NATIONAL EXPOSURE RESEARCH LABORATORY  
RESEARCH TRIANGLE PARK, NC 27711

OFFICE OF  
RESEARCH AND DEVELOPMENT

October 4, 2018

Mr. Clark Freise, Assistant Commissioner  
New Hampshire Department of Environmental Services (NHDES)  
29 Hazen Drive  
P.O. Box 95  
Concord, New Hampshire 03301

Dear Mr. Freise:

I am pleased to provide the attached 3<sup>rd</sup> and 4<sup>th</sup> report from our ongoing collaborative technical support to NHDES assisting with concern over PFAS environmental contamination associated with manufacturing sites. These reports are in response to your request of June 22, 2017 asking for laboratory assistance analyzing per- and polyfluoroalkyl substances (PFAS) in environmental samples. The enclosed Report #3 provides results for surface and ground water samples. Report #4 provides the results of stack sampling.

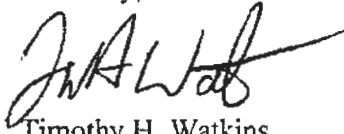
It is our understanding that this information was requested by NHDES to help in your ongoing investigation into the presence of per- and polyfluorinated alkyl substances (PFAS) in the environment near manufacturing facilities of interest. This request relates to our research capabilities and interests applying targeted and non-targeted analysis methods for discovery of the nature and extent of PFAS environmental occurrence that may be potentially associated with industrial releases. EPA continues to develop analytical methods for many PFAS compounds in various media including some of those included in this report. We are providing the results of our analysis as they become available.

In this report, we do not interpret exposure or risk from these values. EPA does not currently have health-based standards, toxicity factors, or associated risk levels for PFAS, other than perfluorooctanoic acid (PFOA), perfluorocatane sulfonate (PFOS), and perfluorobutanesulfonic acid (PFBS). While the data provided in the attached reports indicate the presence of PFAS in water samples, no conclusions can be made related to human or environmental exposure and risk.

Thank you for inviting us to be part of this effort that helps to further both EPA's and New Hampshire's understanding of an important issue in the state. This is just one of many Agency efforts that demonstrates EPA's commitment to cooperative federalism.

If you have any questions or concerns, do not hesitate to contact me at (919) 541-2107 or via email at [watkins.tim@epa.gov](mailto:watkins.tim@epa.gov) or Tim Buckley at (919) 541-2454 or via email at [buckley.timothy@epa.gov](mailto:buckley.timothy@epa.gov). I look forward to our continued work together.

Sincerely,

A handwritten signature in black ink, appearing to read 'Tim H. Watkins', with a stylized flourish at the end.

Timothy H. Watkins  
Director  
National Exposure Research Laboratory  
Office of Research and Development

Enclosure

CC: Meghan Cassidy, USEPA, Region 1  
Deb Szaro, USEPA, Region 1  
Jeff Morris, USEPA OPPT  
Betsy Behl, USEPA, OW  
Peter Grevatt, USEPA, OW  
Andy Gillespie, USEPA, ORD  
Timothy Buckley, USEPA, ORD  
Cindy Sonich-Mullen, USEPA, ORD

**ORD Report #4: Technical Support to New Hampshire -  
Initial Results of CIMS and TO-15 Measurements in Stack Emission SUMMA Canisters**

**Date:** October 4, 2018

**Report Team**

- **Laboratory Chemists:** Theran Riedel, Ingrid George
- **Quality Assurance Review:** Sania Tong-Argao, Margie Vazquez, Libby Nessley and Brittany Stuart
- **Management Coordination and Review:** Myriam Medina-Vera, Adam Biales, Surender Kaushik, Brian Schumacher, Jacky Rosati, Richard Shores, Brian Gullett, Lara Phelps and Timothy Buckley
- **Report Preparation:** John Offenberger and Jeff Ryan

**Summary of Methods and Results**

New Hampshire Department of Environmental Services (NHDES), in coordination with US EPA Region 1, requested technical support from US Environmental Protection Agency, Office of Research and Development (ORD) in analyzing per- and polyfluoroalkyl substances (PFAS) in stack emission samples from an industrial site within the state. NHDES assumed responsibility for the coordination of sample collection protocols with the industry. ORD was responsible for chemical analysis. We are hereby providing the initial results of whole air samples collected in stainless steel, electropolished ("SUMMA" polished), passivated 6-liter canisters over the period of 26 April 2018 to 01 May 2018.

This report includes non-targeted analysis results conducted by high resolution chemical ionization mass spectrometry (CIMS), as well as more conventional TO-15 analysis for specific, volatile organic hazardous air pollutants (HAPs) using unit mass resolution (i.e., 'low res') gas chromatography-mass spectrometry (GC-MS). This work is an initial attempt to adapt ambient SUMMA canisters to collect whole air, stack exhaust gases for laboratory analysis of PFAS by CIMS, as well as by TO-15, and full-scan, low resolution mass spectrometry. The performance of adapting TO-15 to stack emissions is not currently known. Any use of quantitative results should acknowledge such limitations.

Due to this being the first time SUMMA canister sampling for PFAS compounds has been performed on/with stack emissions, the CIMS analyses are limited to tentative identifications, rather than quantification. After tentative identifications are confirmed, additional work will be needed to assess sample collection, transport, handling, and analysis impacts on measurements prior to the development of quantitative analyses. Additionally, non-targeted analysis differs from the more traditional, targeted analysis in that chemical identification and quantification does not have the benefit of being based on authentic standards. As such, one can expect greater uncertainty, both in terms of identification and concentration estimates through a non-targeted analysis. Some of the differences between the analysis methods are summarized in **Table 1**.

The PFAS reported here were identified using non-targeted analysis methods described within an approved quality assurance project plan (QAPP)<sup>1</sup> and addendum<sup>2</sup>. These non-targeted methods are also generally described in Rankin et al., 2015.<sup>3</sup> The Chemical Ionization Mass Spectrometer (CIMS - Aerodyne Research, Inc, Billerica, MA) is a moderately high resolution, direct air inlet, high time resolution mass spectrometer,<sup>4, 5</sup> that has not previously been applied to non-targeted analysis of PFAS in air samples.

As stated, this study adapted ambient, whole-air SUMMA sampling to the plant's stack emissions. In brief, multiple evacuated SUMMA canisters were filled on-site to approximately 7 pounds per square inch absolute (psia), then transported to Research Triangle Park, North Carolina (RTP, NC) for analysis. Incomplete filling during sampling was performed in order to help prevent condensation of water induced by temperature changes during transport to RTP, NC. Upon receipt by ORD, each canister was pressurized to approximately 50 psia with clean air. Actual initial and final pressures were recorded in laboratory notebooks. Samples were analyzed first by chemical ionization mass spectrometry (CIMS) using iodide (I-) as the reagent gas until the pressure inside the canister reached 19 psia, at which point the canister was analyzed according to TO-15, with the additional targeted hydrocarbons of the Photochemical Assessment Monitoring Station list (PAMS). Canisters were then re-analyzed by a full-scan, low mass resolution gas chromatography - mass spectrometry (GC/MS) analysis. The TO-15 analysis can be understood as a targeted analysis of gas phase Hazardous Air Pollutants (HAPs). The subsequent full-scan analysis is similar to the non-targeted analysis presented above and in earlier work, yet the use of low mass resolution GC-MS work results in data that may be of insufficient quality to perform high resolution non-targeted data analysis.

The CIMS non-targeted PFAS compounds are tentatively identified based on the combination of high-resolution mass spectral data (i.e., response vs.  $m/Q$ ), along with interpretation of spectral patterns, such as the presence, or absence, of a series of analogues with the addition of 50 (or 100)  $m/Q$  which is consistent with the addition of CF<sub>2</sub> (or C<sub>2</sub>F<sub>4</sub>) to the backbone of the

---

<sup>1</sup> Strynar, M.; Washington, J.; Lindstrom, A.; Henderson, W. 2017. Quality Assurance Project Plan: Non-Targeted Analyses of Per- and Polyfluoroalkyl Substances (PFAS) for New Hampshire Department of Environmental Services (NHDES). D-EMMD-PHCB-015-QAPP-01.

<sup>2</sup> Offenber, J.H.. Addendum to Quality Assurance Project Plan: Non-Targeted Analyses of Per- and Polyfluoroalkyl Substances (PFAS) for New Hampshire Department of Environmental Services (NHDES). D-EMMD-PHCB-015-QAPP-01-01.

<sup>3</sup> K. Rankin, S. A. Mabury, T. M. Jenkins, J. W. Washington, A North American and global survey of perfluoroalkyl substances in surface Soil: Distribution patterns and mode of occurrence. *Chemosphere* 161, 333-341 (2015).

<sup>4</sup> Lee, B. H.; Lopez-Hilfiker, F. D.; Mohr, C.; Kurtén, T.; Worsnop, D. R.; Thornton, J. A., An Iodide-Adduct High-Resolution Time-of-Flight Chemical-Ionization Mass Spectrometer: Application to Atmospheric Inorganic and Organic Compounds. *Environmental Science & Technology* **2014**, 48 (11), 6309-6317.

<sup>5</sup> Lopez-Hilfiker, F. D.; Iyer, S.; Mohr, C.; Lee, B. H.; D'Ambro, E. L.; Kurtén, T.; Thornton, J. A., Constraining the sensitivity of iodide adduct chemical ionization mass spectrometry to multifunctional organic molecules using the collision limit and thermodynamic stability of iodide ion adducts. *Atmos. Meas. Tech.* **2016**, 9 (4), 1505-1512.

molecule. Without the benefit of known authentic standards, identifications are effectively limited to library search matches, as well as agreement between estimates of molecular mass from empirical formulae, and measured  $m/Q$  by the instrument.

After CIMS analysis, samples were also analyzed by GC/MS according to TO-15 using Selective Ion Monitoring (SIM), then again by GC-MS, under the same chromatographic conditions, operating in full scan mode over a range of  $m/Q$  of 31 to 500. The addition of chromatography and higher energy ionization (70 eV Electron Impact) may allow for additional comparative analysis between the full scan GC-MS (low resolution full scan) and CIMS. Both CIMS and GC-MS analyses were performed on all samples, and process blanks in an identical manner. For all following results, ions (i.e., compounds) observed across all three replicate canisters are indicated in the following tables, corresponding with the chemical analysis performed.

The CIMS-based non-targeted analysis tentatively identified twelve (12) PFAS compounds in the SUMMA canisters (**Table 2**). Some ambiguity remains in the assignment of the tentative identifications, largely due to the combination of soft (i.e. non-fragmentary) ionization with no chromatographic separation in the CIMS technique. For example, two PFAS compounds (6:2 telomer alcohol 364.10 Th, and the C7 perfluoro-carboxylic acid 364.10 Th) have nearly identical molecular weights, and are subsequently observed in the CIMS at nearly indistinguishable  $m/Q$ . Given the resolution of the CIMS instrument ( $\sim 3000$ ), it is not possible to definitively identify which of the two compounds is present ( $x:2$  telomer alcohol, or the corresponding  $x + 1$  perfluoro-carboxylic acid). At present we cannot determine the empirical formulae, and therefore, have not specified a 'most likely' identification nor corresponding CAS number.

The GC-MS targeted SIM analysis of SUMMA canisters identified 27, 42 and 38 non-PFAS compounds in the samples from the MS, MA and QX towers, respectively. Compounds identified are listed in **Table 3**. Across all 9 SUMMA canisters representing non-controlled stack emissions (i.e., from MS Tower, MA Tower, and QX Tower Inlet sampling), the following compounds were observed in all canisters: propylene, propane, chloromethane, isobutane, 1-butene, ethanol, acrolein, acetone, iso-pentane, isopropyl alcohol, 1-pentene, isoprene, vinyl acetate, 2-butanone, 1-hexene, tetrahydrofuran, 2,4-dimethylpentane, benzene, 4-methyl-2-pentanone, toluene, and dodecane. Up to 118 gas phase compounds were observed across all nine samples using the TO-15 (plus PAMS compounds) method.

Comparison of pre- and post- control device implementation on the QX tower showed a small reduction in the number of compounds identified in all 3 of the post-control device canisters. However, this reduction cannot be interpreted regarding the effectiveness of the control device. Impacts, if any, of the control devices are best assessed through direct comparison of quantitative results.

Measured concentrations of the TO-15 (plus PAMS) compounds are given in **Table 4** through **Table 7**. All measured concentrations are reported as parts per billion by volume (ppbV). In all four of these tables, there are several data descriptors (often called 'flags') to identify a) the presence of contamination which leads to suspect quantitation, b) a compound coelution problem which leads to unreliable quantitation, c) a compound which was above the calibration range,



from which an estimated value is based on highest calibration point (i.e., presented as greater than the highest calibration point). Compounds below the method detection limit (MDL) are reported as < method quantitation limit (MQL). Reported values are blank corrected and normalized for dilution of the original sample by the addition of clean air. **Table 4** presents concentrations measured in SUMMA canisters collected on the MS Tower, while **Table 5** lists concentrations measured in samples collected on the MA Tower. **Table 6** includes measured concentrations of compounds in the SUMMA canisters collected on both the inlet and outlet of the control device on the QX Tower. **Table 7** includes results from three ambient samples as well as the single field blank canister.

Data analysis of the full-scan GC-MS analysis of these same canisters is not yet complete. The additional analyses may provide insight into the relative contributions of the x:2 fluoro-telomer alcohols, relative to the corresponding x+1 perfluoro-carboxylic acids. The fragmentation patterns of the more energetic ionization technique, as well as the potential for chromatographic separation of analytes, may provide multiple confirmatory results when compared with authentic standards. These evaluations, along with initial laboratory studies of the stability of select PFAS compounds in SUMMA canisters, is currently being performed by ORD in RTP, NC.

**Table 1.** Overview of Collection and Analysis Methods used for SUMMA canister analysis. For comparison, LC-TOF MS and LC-HRMS, used in prior work, are also included.

<b>Analysis Method</b>	<b>Mass Resolution</b>	<b>Chromatography</b>	<b>m/Q range</b>	<b>Identification</b>	<b>Negative Mass Defect</b>	<b>Quantification</b>
CIMS	Moderately High 0.00X	No	1-1000	I <sup>-</sup> Adducts  Formulae only  No Fragmentation	Yes	No, not yet
TO-15	Low (unit mass)	Yes	Selected Ion Monitoring	Confirmed with Authentic Standards  where possible	No	Yes
GC/MS	Low (unit mass)	Yes	31-500	Peak ID,  Library search,  Diagnostic fragments	No	No
LC-TOF MS  LC-HRMS (Orbitrap)	High 0.000x	Yes	e.g.  100-1000	PFAS Library Standards	Yes	Yes, for many

**Table 2.** PFAS ions observed by CIMS analysis of SUMMA canister stack air samples collected from a New Hampshire manufacturing facility. Concentration values are expressed in arbitrary units.

**PRELIMINARY DATA**			Probable PFAS Composition (mean signal, dilution normalized)											
Can ID	Sample Type	Dilution Factor	C3FH5O2	C5F3H9O3	C6F2H6O2	C6F9H5O	C7F6H10O2	C7F11H5O	C7F13H3O	C8F7H5O4	C8F11H7O2	C8F13H5O	C9FH7O2	C10F13H9O2
			92.0274	174.050	148.033	264.019	240.058	314.016	349.997	298.007	344.027	364.013	166.043	408.039
background 1	system blank	1	532	21	16	17	9	9	6	2	3	16	39	5
RK9	ambient (inside facility)	4.6	12296	113	48	47	71	45	40	29	27	332	757	35
5	ambient (lower roof)	6.1	5445	148	237	38	108	59	38	54	29	102	407	66
794	ambient (upper roof)	10.4	4656	225	74	135	543	181	66	50	41	348	1273	53
709	ambient? (Field Blank)	1	529	17	5	9	16	9	5	4	7	16	59	6
755	MA tower (Run 1)	10.2	42844	886	1185	152	611	168	102	59	56	171	5140	139
751	MA tower (Run 2)	10.2	57354	1145	1181	234	483	244	95	117	33	230	5400	72
262	MA tower (Run 3)	9.1	66907	1028	452	306	464	180	133	53	85	846	1603	108
68	MS tower (Run 1)	9.7	21290	612	476	77	425	110	160	72	50	1849	2863	296
700	MS tower (Run 2)	9.4	32577	364	463	115	558	117	82	50	74	242	6703	29
744	MS tower (Run 3)	9.4	37052	335	111	157	407	159	53	63	76	207	3079	61
721	QX tower inlet (Run 1)	10.2	37442	1200	1144	294	748	321	10213	1612	628	44901	7520	793
176	QX tower inlet (Run 2)	9.4	32334	1521	948	280	706	272	9315	1605	430	43836	7291	945
A378	QX tower inlet (Run 3)	9.5	12112	446	535	130	715	196	313	127	78	2015	2786	217
321	QX tower outlet (Run 1)	9.1	30660	501	457	313	430	171	4952	790	332	24015	3852	152
2045	QX tower outlet (Run 2)	8.9	20723	1195	687	579	393	585	10556	1230	958	85658	2251	539
793	QX tower outlet (Run 3)	8.2	52629	598	678	96	428	124	171	70	81	1914	2029	295
background 2	system blank	1	889	33	32	11	22	9	7	9	8	17	51	10

**Table 3.** Compounds identified using selected ion monitoring GC/MS – **Method:** TO-15.**Summary of Compounds Present in TO-15 Samples**

Yellow Highlight indicates compound detected in all 3 Runs			
MS Tower TO-15 Target Compounds	MA Tower TO-15 Target Compounds	QX Tower Inlet TO-15 Target Compounds	QX Tower Outlet TO-15 Target Compounds
Propylene	Propylene	Propylene	Propylene
Propane	Propane	Propane	Propane
Dichlorodifluoromethane	Dichlorodifluoromethane	Dichlorodifluoromethane	Dichlorodifluoromethane
Chloromethane	Chloromethane	Chloromethane	Chloromethane
Isobutane	Isobutane	Isobutane	Isobutane
Dichlorotetrafluoroethane	Dichlorotetrafluoroethane	Dichlorotetrafluoroethane	Dichlorotetrafluoroethane
Vinyl Chloride	Vinyl Chloride	Vinyl Chloride	Vinyl Chloride
1-Butene	1-Butene	1-Butene	1-Butene
1,3-Butadiene	1,3-Butadiene	1,3-Butadiene	1,3-Butadiene
Butane	Butane	Butane	Butane
trans-2-butene	trans-2-butene	trans-2-butene	trans-2-butene
Bromomethane	Bromomethane	Bromomethane	Bromomethane
cis-2-butene	cis-2-butene	cis-2-butene	cis-2-butene
Chloroethane	Chloroethane	Chloroethane	Chloroethane
Ethanol	Ethanol	Ethanol	Ethanol
Vinyl Bromide	Vinyl Bromide	Vinyl Bromide	Vinyl Bromide
Acetonitrile	Acetonitrile	Acetonitrile	Acetonitrile
Acrolein	Acrolein	Acrolein	Acrolein
Acetone	Acetone	Acetone	Acetone
iso-Pentane	iso-Pentane	iso-Pentane	iso-Pentane
Trichlorofluoromethane	Trichlorofluoromethane	Trichlorofluoromethane	Trichlorofluoromethane
Isopropyl Alcohol	Isopropyl Alcohol	Isopropyl Alcohol	Isopropyl Alcohol
1-Pentene	1-Pentene	1-Pentene	1-Pentene
Acrylonitrile	Acrylonitrile	Acrylonitrile	Acrylonitrile
n-Pentane	n-Pentane	n-Pentane	n-Pentane
Isoprene	Isoprene	Isoprene	Isoprene
trans-2-pentene	trans-2-pentene	trans-2-pentene	trans-2-pentene
cis-2-pentene	cis-2-pentene	cis-2-pentene	cis-2-pentene
Tert-Butanol	Tert-Butanol	Tert-Butanol	Tert-Butanol
1,1-Dichloroethene	1,1-Dichloroethene	1,1-Dichloroethene	1,1-Dichloroethene
Methylene Chloride	Methylene Chloride	Methylene Chloride	Methylene Chloride
3-Chloro-1-Propene	3-Chloro-1-Propene	3-Chloro-1-Propene	3-Chloro-1-Propene

MS Tower	MA Tower	QX Tower Inlet	QX Tower Outlet
TO-15 Target Compounds	TO-15 Target Compounds	TO-15 Target Compounds	TO-15 Target Compounds
1,1,2-Trichloro-1,2,2-trifluoroethane	1,1,2-Trichloro-1,2,2-trifluoroethane	1,1,2-Trichloro-1,2,2-trifluoroethane	1,1,2-Trichloro-1,2,2-trifluoroethane
Carbon Disulfide	Carbon Disulfide	Carbon Disulfide	Carbon Disulfide
2,2-Dimethylbutane	2,2-Dimethylbutane	2,2-Dimethylbutane	2,2-Dimethylbutane
trans-1,2-Dichloroethene	trans-1,2-Dichloroethene	trans-1,2-Dichloroethene	trans-1,2-Dichloroethene
Cyclopentane	Cyclopentane	Cyclopentane	Cyclopentane
2,3-Dimethylbutane	2,3-Dimethylbutane	2,3-Dimethylbutane	2,3-Dimethylbutane
1,1-Dichloroethane	1,1-Dichloroethane	1,1-Dichloroethane	1,1-Dichloroethane
Methyl-t-Butyl-Ether	Methyl-t-Butyl-Ether	Methyl-t-Butyl-Ether	Methyl-t-Butyl-Ether
Vinyl Acetate	Vinyl Acetate	Vinyl Acetate	Vinyl Acetate
2-Methylpentane	2-Methylpentane	2-Methylpentane	2-Methylpentane
2-Butanone	2-Butanone	2-Butanone	2-Butanone
3-Methylpentane	3-Methylpentane	3-Methylpentane	3-Methylpentane
2-Chloroprene	2-Chloroprene	2-Chloroprene	2-Chloroprene
1-Hexene	1-Hexene	1-Hexene	1-Hexene
cis-1,2-Dichloroethene	cis-1,2-Dichloroethene	cis-1,2-Dichloroethene	cis-1,2-Dichloroethene
Diisopropyl ether	Diisopropyl ether	Diisopropyl ether	Diisopropyl ether
Ethyl Acetate	Ethyl Acetate	Ethyl Acetate	Ethyl Acetate
n-Hexane	n-Hexane	n-Hexane	n-Hexane
Chloroform	Chloroform	Chloroform	Chloroform
Tetrahydrofuran	Tetrahydrofuran	Tetrahydrofuran	Tetrahydrofuran
Ethyl Tert-Butyl Ether	Ethyl Tert-Butyl Ether	Ethyl Tert-Butyl Ether	Ethyl Tert-Butyl Ether
Methylcyclopentane	Methylcyclopentane	Methylcyclopentane	Methylcyclopentane
1,2-Dichloroethane	1,2-Dichloroethane	1,2-Dichloroethane	1,2-Dichloroethane
2,4-Dimethylpentane	2,4-Dimethylpentane	2,4-Dimethylpentane	2,4-Dimethylpentane
1,1,1-Trichloroethane	1,1,1-Trichloroethane	1,1,1-Trichloroethane	1,1,1-Trichloroethane
Benzene	Benzene	Benzene	Benzene
Carbon Tetrachloride	Carbon Tetrachloride	Carbon Tetrachloride	Carbon Tetrachloride
Cyclohexane	Cyclohexane	Cyclohexane	Cyclohexane
2-Methylhexane	2-Methylhexane	2-Methylhexane	2-Methylhexane
2,3-Dimethylpentane	2,3-Dimethylpentane	2,3-Dimethylpentane	2,3-Dimethylpentane
Tert Amyl Methyl Ether	Tert Amyl Methyl Ether	Tert Amyl Methyl Ether	Tert Amyl Methyl Ether
3-methylhexane	3-methylhexane	3-methylhexane	3-methylhexane
1,2-Dichloropropane	1,2-Dichloropropane	1,2-Dichloropropane	1,2-Dichloropropane
Bromodichloromethane	Bromodichloromethane	Bromodichloromethane	Bromodichloromethane
1,4-Dioxane	1,4-Dioxane	1,4-Dioxane	1,4-Dioxane
Trichloroethene	Trichloroethene	Trichloroethene	Trichloroethene
Isooctane	Isooctane	Isooctane	Isooctane
Methyl Methacrylate	Methyl Methacrylate	Methyl Methacrylate	Methyl Methacrylate
Heptane	Heptane	Heptane	Heptane
cis-1,3-Dichloropropene	cis-1,3-Dichloropropene	cis-1,3-Dichloropropene	cis-1,3-Dichloropropene
4-Methy-2-Pentanone	4-Methy-2-Pentanone	4-Methy-2-Pentanone	4-Methy-2-Pentanone



MS Tower	MA Tower	QX Tower Inlet	QX Tower Outlet
TO-15 Target Compounds	TO-15 Target Compounds	TO-15 Target Compounds	TO-15 Target Compounds
Methylcyclohexane	Methylcyclohexane	Methylcyclohexane	Methylcyclohexane
trans-1,3-Dichloropropene	trans-1,3-Dichloropropene	trans-1,3-Dichloropropene	trans-1,3-Dichloropropene
1,1,2-Trichloroethane	1,1,2-Trichloroethane	1,1,2-Trichloroethane	1,1,2-Trichloroethane
2,3,4-Trimethylpentane	2,3,4-Trimethylpentane	2,3,4-Trimethylpentane	2,3,4-Trimethylpentane
Toluene	Toluene	Toluene	Toluene
2-Methylheptane	2-Methylheptane	2-Methylheptane	2-Methylheptane
Dibromochloromethane	Dibromochloromethane	Dibromochloromethane	Dibromochloromethane
3-Methylheptane	3-Methylheptane	3-Methylheptane	3-Methylheptane
1,2-Dibromoethane	1,2-Dibromoethane	1,2-Dibromoethane	1,2-Dibromoethane
Octane	Octane	Octane	Octane
Tetrachloroethene	Tetrachloroethene	Tetrachloroethene	Tetrachloroethene
1,1,1,2-Tetrachloroethane	1,1,1,2-Tetrachloroethane	1,1,1,2-Tetrachloroethane	1,1,1,2-Tetrachloroethane
Chlorobenzene	Chlorobenzene	Chlorobenzene	Chlorobenzene
Ethylbenzene	Ethylbenzene	Ethylbenzene	Ethylbenzene
m-Xylene	m-Xylene	m-Xylene	m-Xylene
p-Xylene	p-Xylene	p-Xylene	p-Xylene
Bromoform	Bromoform	Bromoform	Bromoform
Styrene	Styrene	Styrene	Styrene
1,1,2,2-Tetrachloroethane	1,1,2,2-Tetrachloroethane	1,1,2,2-Tetrachloroethane	1,1,2,2-Tetrachloroethane
o-Xylene	o-Xylene	o-Xylene	o-Xylene
Nonane	Nonane	Nonane	Nonane
Cumene	Cumene	Cumene	Cumene
n-Propylbenzene	n-Propylbenzene	n-Propylbenzene	n-Propylbenzene
m-Ethyltoluene	m-Ethyltoluene	m-Ethyltoluene	m-Ethyltoluene
1,3,5-Trimethylbenzene	1,3,5-Trimethylbenzene	1,3,5-Trimethylbenzene	1,3,5-Trimethylbenzene
1,2,4-Trimethylbenzene	1,2,4-Trimethylbenzene	1,2,4-Trimethylbenzene	1,2,4-Trimethylbenzene
Tert-Butyl Benzene	Tert-Butyl Benzene	Tert-Butyl Benzene	Tert-Butyl Benzene
1-Ethyl-4-Methyl Benzene	1-Ethyl-4-Methyl Benzene	1-Ethyl-4-Methyl Benzene	1-Ethyl-4-Methyl Benzene
o-Ethyltoluene	o-Ethyltoluene	o-Ethyltoluene	o-Ethyltoluene
1,3-Dichlorobenzene	1,3-Dichlorobenzene	1,3-Dichlorobenzene	1,3-Dichlorobenzene
1,4-Dichlorobenzene	1,4-Dichlorobenzene	1,4-Dichlorobenzene	1,4-Dichlorobenzene
n-Decane	n-Decane	n-Decane	n-Decane
Sec-Butyl Benzene	Sec-Butyl Benzene	Sec-Butyl Benzene	Sec-Butyl Benzene
1,2,3-Trimethylbenzene	1,2,3-Trimethylbenzene	1,2,3-Trimethylbenzene	1,2,3-Trimethylbenzene
1,2-Dichlorobenzene	1,2-Dichlorobenzene	1,2-Dichlorobenzene	1,2-Dichlorobenzene
o-Cymene	o-Cymene	o-Cymene	o-Cymene
1,3-Diethylbenzene	1,3-Diethylbenzene	1,3-Diethylbenzene	1,3-Diethylbenzene
1,2-Diethylbenzene	1,2-Diethylbenzene	1,2-Diethylbenzene	1,2-Diethylbenzene
n-Butyl Benzene	n-Butyl Benzene	n-Butyl Benzene	n-Butyl Benzene
Undecane	Undecane	Undecane	Undecane
1,2,4-Trichlorobenzene	1,2,4-Trichlorobenzene	1,2,4-Trichlorobenzene	1,2,4-Trichlorobenzene
Naphthalene	Naphthalene	Naphthalene	Naphthalene

MS Tower	MA Tower	QX Tower Inlet	QX Tower Outlet
TO-15 Target Compounds	TO-15 Target Compounds	TO-15 Target Compounds	TO-15 Target Compounds
Dodecane	Dodecane	Dodecane	Dodecane
Hexachlorobutadiene	Hexachlorobutadiene	Hexachlorobutadiene	Hexachlorobutadiene

**Table 4.** Measured concentrations of TO-15 (plus PAMS) compounds in MS Tower SUMMA canisters.

MS Tower				
Can ID	368	700	744	
Location	MS Tower	MS Tower	MS Tower	
Run Number	Run 1	Run 2	Run 3	
Date	4/26/2018	4/27/2018	4/27/2018	

**Data Descriptors**

- Contamination present. Quantitation suspect.
- Compound coelution problem. Quantitation not reliable.
- Above calibration range. Estimated value based on highest calibration point.
- Compounds below 3 x MDL are reported as '< MQL'
- Reported values are blank corrected

Target Compounds	Conc. ppbv	Conc. ppbv	Conc. ppbv
Propylene	139.85	107.06	83.88
Propane	73.75	61.77	33.24
Dichlorodifluoromethane	0.29	0.35	0.35
Chloromethane	2.14	2.53	0.94
Isobutane	101.63	48.29	21.61
Dichlorotetrafluoroethane	< MQL	< MQL	< MQL
Vinyl Chloride	< MQL	< MQL	< MQL
1-Butene	101.84	73.85	57.98
1,3-Butadiene	6.08	4.18	3.47
Butane	0.87	1.85	0.74
trans-2-butene	1.45	1.77	< MQL
Bromomethane	< MQL	< MQL	< MQL
cis-2-butene	1.71	< MQL	< MQL
Chloroethane	< MQL	< MQL	< MQL
Ethanol	56.95	24.50	15.05
Vinyl Bromide	< MQL	< MQL	< MQL
Acetonitrile	12.75	13.98	3.72
Acrolein	182.90	97.20	57.61
Can ID	368	700	744



	Location	MS Tower	MS Tower	MS Tower
	Run Number	Run 1	Run 2	Run 3
	Date	4/26/2018	4/27/2018	4/27/2018
Target Compounds		Conc.	Conc.	Conc.
		ppbv	ppbv	ppbv
Acetone		>413.776	>404.709	>410.84
iso-Pentane		17.77	41.61	8.88
Trichlorofluoromethane		< MQL	< MQL	< MQL
Isopropyl Alcohol		46.78	36.05	35.94
1-Pentene		2.32	1.42	0.82
Acrylonitrile		0.78	1.32	< MQL
n-Pentane		1.28	3.03	0.88
Isoprene		0.52	0.48	0.28
trans-2-pentene		< MQL	< MQL	< MQL
cis-2-pentene		< MQL	< MQL	< MQL
Tert-Butanol		1.54	1.04	< MQL
1,1-Dichloroethene		< MQL	< MQL	< MQL
Methylene Chloride		< MQL	< MQL	< MQL
3-Chloro-1-Propene		< MQL	< MQL	< MQL
1,1,2-Trichloro-1,2,2-trifluoroethane		< MQL	< MQL	< MQL
Carbon Disulfide		1.43	1.10	0.82
2,2-Dimethylbutane		< MQL	< MQL	< MQL
trans-1,2-Dichloroethene		< MQL	< MQL	< MQL
Cyclopentane		16.87	10.25	< MQL
2,3-Dimethylbutane		< MQL	< MQL	< MQL
1,1-Dichloroethane		< MQL	< MQL	< MQL
Methyl-t-Butyl-Ether		< MQL	< MQL	< MQL
Vinyl Acetate		45.72	34.31	26.88
2-Methylpentane		< MQL	< MQL	< MQL
2-Butanone		150.61	53.48	43.89
3-Methylpentane		0.61	0.91	< MQL
2-Chloroprene		< MQL	< MQL	< MQL
1-Hexene		2.05	1.21	0.40
cis-1,2-Dichloroethene		< MQL	< MQL	< MQL
Diisopropyl ether		< MQL	< MQL	< MQL
Ethyl Acetate		< MQL	< MQL	< MQL
n-Hexane		< MQL	0.66	< MQL
Chloroform		< MQL	< MQL	< MQL
Tetrahydrofuran		3.37	3.74	2.48
Ethyl Tert-Butyl Ether		< MQL	< MQL	< MQL
Methylcyclopentane		< MQL	0.23	< MQL
	Can ID	368	700	744

	Location	MS Tower	MS Tower	MS Tower
	Run Number	Run 1	Run 2	Run 3
	Date	4/26/2018	4/27/2018	4/27/2018
Target Compounds	Conc.	Conc.	Conc.	
	ppbv	ppbv	ppbv	
1,2-Dichloroethane	< MQL	< MQL	< MQL	
2,4-Dimethylpentane	0.74	0.43	0.23	
1,1,1-Trichloroethane	< MQL	< MQL	< MQL	
Benzene	110.06	21.07	5.21	
Carbon Tetrachloride	< MQL	< MQL	< MQL	
Cyclohexane	0.81	1.53	0.64	
2-Methylhexane	0.50	1.36	< MQL	
2,3-Dimethylpentane	< MQL	< MQL	< MQL	
Tert Amyl Methyl Ether	< MQL	< MQL	< MQL	
3-methylhexane	3.38	1.56	< MQL	
1,2-Dichloropropane	< MQL	< MQL	< MQL	
Bromodichloromethane	< MQL	< MQL	< MQL	
1,4-Dioxane	16.22	10.98	< MQL	
Trichloroethene	< MQL	< MQL	< MQL	
Isooctane	< MQL	< MQL	< MQL	
Methyl Methacrylate	< MQL	< MQL	< MQL	
Heptane	< MQL	1.59	< MQL	
cis-1,3-Dichloropropene	< MQL	< MQL	< MQL	
4-Methy-2-Pentanone	204.92	121.73	102.23	
Methylcyclohexane	0.38	1.30	< MQL	
trans-1,3-Dichloropropene	< MQL	< MQL	< MQL	
1,1,2-Trichloroethane	< MQL	< MQL	< MQL	
2,3,4-Trimethylpentane	< MQL	0.26	< MQL	
Toluene	21.40	49.50	4.59	
2-Methylheptane	< MQL	0.52	< MQL	
Dibromochloromethane	< MQL	< MQL	< MQL	
3-Methylheptane	< MQL	1.93	1.02	
1,2-Dibromoethane	< MQL	< MQL	< MQL	
Octane	< MQL	0.62	< MQL	
Tetrachloroethene	< MQL	< MQL	< MQL	
1,1,1,2-Tetrachloroethane	< MQL	< MQL	< MQL	
Chlorobenzene	< MQL	< MQL	< MQL	
Ethylbenzene	0.45	0.58	< MQL	
m-Xylene	< MQL	0.81	< MQL	
p-Xylene	< MQL	< MQL	< MQL	
Bromoform	< MQL	< MQL	< MQL	
Can ID	368	700	744	

	Location	MS Tower	MS Tower	MS Tower
	Run Number	Run 1	Run 2	Run 3
	Date	4/26/2018	4/27/2018	4/27/2018
Target Compounds	Conc.	Conc.	Conc.	
	ppbv	ppbv	ppbv	
Styrene	< MQL	< MQL	< MQL	
1,1,2,2-Tetrachloroethane	< MQL	< MQL	< MQL	
o-Xylene	< MQL	< MQL	< MQL	
Nonane	1.55	1.31	< MQL	
Cumene	< MQL	0.20	< MQL	
n-Propylbenzene	< MQL	0.37	< MQL	
m-Ethyltoluene	< MQL	0.41	< MQL	
1,3,5-Trimethylbenzene	< MQL	< MQL	< MQL	
1,2,4-Trimethylbenzene	< MQL	< MQL	< MQL	
Tert-Butyl Benzene	< MQL	< MQL	< MQL	
1-Ethyl-4-Methyl Benzene	< MQL	< MQL	< MQL	
o-Ethyltoluene	< MQL	< MQL	< MQL	
1,3-Dichlorobenzene	< MQL	< MQL	< MQL	
1,4-Dichlorobenzene	< MQL	< MQL	< MQL	
n-Decane	< MQL	< MQL	< MQL	
Sec-Butyl Benzene	< MQL	< MQL	< MQL	
1,2,3-Trimethylbenzene	< MQL	< MQL	< MQL	
1,2-Dichlorobenzene	< MQL	< MQL	< MQL	
o-Cymene	< MQL	< MQL	< MQL	
1,3-Diethylbenzene	< MQL	0.34	< MQL	
1,2-Diethylbenzene	< MQL	0.39	< MQL	
n-Butyl Benzene	< MQL	< MQL	< MQL	
Undecane	0.90	1.28	< MQL	
1,2,4-Trichlorobenzene	< MQL	< MQL	< MQL	
Naphthalene	0.80	0.63	< MQL	
Dodecane	36.39	6.67	30.64	
Hexachlorobutadiene	< MQL	< MQL	< MQL	

**Table 5.** Measured concentrations of TO-15 (plus PAMS) compounds in MA Tower SUMMA canisters.

MA Tower				
	Can ID	755	751	262
	Location	MA Tower	MA Tower	MA Tower
	Run Number	Run 1	Run 2	Run 3
	Date	4/26/2018	4/26/2018	4/26/2018
<b>Data Descriptors</b>				
<ul style="list-style-type: none"> <li>Contamination present. Quantitation suspect.</li> <li>Compound coelution problem. Quantitation not reliable.</li> <li>Above calibration range. Estimated value based on highest calibration point.</li> <li>Compounds below 3 x MDL are reported as '&lt; MQL'</li> <li>Reported values are blank corrected</li> </ul>				
Target Compounds	Conc. ppbv	Conc. ppbv	Conc. ppbv	
Propylene	785.39	807.99	713.21	
Propane	42.35	45.75	72.05	
Dichlorodifluoromethane	< MQL	0.30	0.29	
Chloromethane	0.55	0.78	1.01	
Isobutane	63.97	77.06	77.51	
Dichlorotetrafluoroethane	< MQL	< MQL	< MQL	
Vinyl Chloride	< MQL	< MQL	< MQL	
1-Butene	>422.306	>425.27	>382.704	
1,3-Butadiene	22.46	24.92	24.15	
Butane	< MQL	1.35	1.61	
trans-2-butene	1.99	5.72	4.54	
Bromomethane	< MQL	< MQL	< MQL	
cis-2-butene	< MQL	1.47	3.49	
Chloroethane	< MQL	< MQL	< MQL	
Ethanol	52.08	60.82	57.70	
Vinyl Bromide	< MQL	< MQL	< MQL	
Acetonitrile	11.25	16.80	14.67	
Acrolein	>432.824	>435.861	>392.235	
	Can ID	755	751	262

Target Compounds	Location	MA Tower	MA Tower	MA Tower
	Run Number	Run 1	Run 2	Run 3
	Date	4/26/2018	4/26/2018	4/26/2018
	Conc.	Conc.	Conc.	
	ppbv	ppbv	ppbv	
Acetone	>436.869	>439.935	>395.901	
iso-Pentane	5.22	173.42	179.83	
Trichlorofluoromethane	< MQL	< MQL	< MQL	
Isopropyl Alcohol	101.44	110.01	117.79	
1-Pentene	2.37	1.11	1.70	
Acrylonitrile	9.03	7.73	7.23	
n-Pentane	< MQL	0.91	1.05	
Isoprene	3.61	3.81	3.35	
trans-2-pentene	0.56	0.74	0.68	
cis-2-pentene	0.40	0.41	0.39	
Tert-Butanol	4.18	3.34	2.87	
1,1-Dichloroethene	< MQL	< MQL	< MQL	
Methylene Chloride	< MQL	< MQL	< MQL	
3-Chloro-1-Propene	< MQL	0.68	0.55	
1,1,2-Trichloro-1,2,2-trifluoroethane	< MQL	< MQL	< MQL	
Carbon Disulfide	< MQL	< MQL	< MQL	
2,2-Dimethylbutane	< MQL	< MQL	< MQL	
trans-1,2-Dichloroethene	< MQL	< MQL	< MQL	
Cyclopentane	7.37	4.06	1.87	
2,3-Dimethylbutane	1.53	< MQL	< MQL	
1,1-Dichloroethane	< MQL	< MQL	< MQL	
Methyl-t-Butyl-Ether	< MQL	< MQL	< MQL	
Vinyl Acetate	86.92	74.80	74.27	
2-Methylpentane	1.51	< MQL	< MQL	
2-Butanone	203.94	172.34	151.31	
3-Methylpentane	1.71	1.66	0.88	
2-Chloroprene	< MQL	0.17	0.09	
1-Hexene	5.70	5.86	5.67	
cis-1,2-Dichloroethene	< MQL	< MQL	< MQL	
Diisopropyl ether	0.57	0.29	0.45	
Ethyl Acetate	< MQL	< MQL	< MQL	
n-Hexane	0.97	0.87	0.43	
Chloroform	< MQL	< MQL	0.11	
Tetrahydrofuran	15.84	16.94	15.40	
Ethyl Tert-Butyl Ether	< MQL	< MQL	< MQL	
Methylcyclopentane	< MQL	< MQL	< MQL	
1,2-Dichloroethane	< MQL	< MQL	< MQL	
2,4-Dimethylpentane	1.89	2.61	2.65	
Can ID	755	751	262	

	Location	MA Tower	MA Tower	MA Tower
	Run Number	Run 1	Run 2	Run 3
	Date	4/26/2018	4/26/2018	4/26/2018
Target Compounds	Conc.	Conc.	Conc.	
	ppbv	ppbv	ppbv	
1,1,1-Trichloroethane	< MQL	< MQL	< MQL	
Benzene	11.52	11.08	9.88	
Carbon Tetrachloride	< MQL	< MQL	< MQL	
Cyclohexane	< MQL	1.55	1.28	
2-Methylhexane	5.15	0.91	1.87	
2,3-Dimethylpentane	0.84	< MQL	< MQL	
Tert Amyl Methyl Ether	< MQL	< MQL	< MQL	
3-methylhexane	5.93	4.07	4.41	
1,2-Dichloropropane	< MQL	< MQL	< MQL	
Bromodichloromethane	< MQL	< MQL	< MQL	
1,4-Dioxane	18.91	21.63	17.61	
Trichloroethene	< MQL	< MQL	< MQL	
Isooctane	< MQL	0.57	< MQL	
Methyl Methacrylate	< MQL	< MQL	< MQL	
Heptane	5.67	< MQL	< MQL	
cis-1,3-Dichloropropene	< MQL	< MQL	< MQL	
4-Methy-2-Pentanone	>420.217	>423.166	>380.811	
Methylcyclohexane	1.93	1.66	0.86	
trans-1,3-Dichloropropene	< MQL	< MQL	< MQL	
1,1,2-Trichloroethane	< MQL	< MQL	< MQL	
2,3,4-Trimethylpentane	< MQL	0.99	0.70	
Toluene	57.88	73.57	14.32	
2-Methylheptane	0.76	0.88	< MQL	
Dibromochloromethane	< MQL	< MQL	< MQL	
3-Methylheptane	1.54	< MQL	0.93	
1,2-Dibromoethane	< MQL	< MQL	< MQL	
Octane	1.00	1.99	0.63	
Tetrachloroethene	< MQL	< MQL	< MQL	
1,1,1,2-Tetrachloroethane	< MQL	< MQL	< MQL	
Chlorobenzene	< MQL	< MQL	< MQL	
Ethylbenzene	< MQL	0.45	< MQL	
m-Xylene	1.42	2.96	1.58	
p-Xylene	< MQL	1.00	< MQL	
Bromoform	< MQL	< MQL	< MQL	
Styrene	< MQL	< MQL	< MQL	
1,1,2,2-Tetrachloroethane	< MQL	0.75	< MQL	
o-Xylene	< MQL	< MQL	< MQL	
Nonane	3.77	5.58	5.01	
Can ID	755	751	262	

	Location	MA Tower	MA Tower	MA Tower
	Run Number	Run 1	Run 2	Run 3
	Date	4/26/2018	4/26/2018	4/26/2018
Target Compounds	Conc.	Conc.	Conc.	
	ppbv	ppbv	ppbv	
Cumene	< MQL	0.24	0.24	
n-Propylbenzene	< MQL	0.52	0.50	
m-Ethyltoluene	< MQL	0.67	0.56	
1,3,5-Trimethylbenzene	< MQL	0.64	0.70	
1,2,4-Trimethylbenzene	< MQL	< MQL	< MQL	
Tert-Butyl Benzene	< MQL	< MQL	< MQL	
1-Ethyl-4-Methyl Benzene	< MQL	< MQL	< MQL	
o-Ethyltoluene	< MQL	< MQL	< MQL	
1,3-Dichlorobenzene	< MQL	< MQL	< MQL	
1,4-Dichlorobenzene	< MQL	< MQL	< MQL	
n-Decane	< MQL	0.58	0.39	
Sec-Butyl Benzene	< MQL	< MQL	< MQL	
1,2,3-Trimethylbenzene	< MQL	< MQL	< MQL	
1,2-Dichlorobenzene	< MQL	< MQL	< MQL	
o-Cymene	< MQL	< MQL	< MQL	
1,3-Diethylbenzene	< MQL	0.51	0.41	
1,2-Diethylbenzene	< MQL	0.92	0.68	
n-Butyl Benzene	< MQL	< MQL	< MQL	
Undecane	1.60	2.47	1.86	
1,2,4-Trichlorobenzene	< MQL	< MQL	< MQL	
Naphthalene	0.88	0.86	0.60	
Dodecane	33.32	>394.967	>355.434	
Hexachlorobutadiene	< MQL	< MQL	< MQL	



**Table 6.** Measured concentrations of TO-15 (plus PAMS) compounds in QX tower Inlet and Outlet SUMMA canisters.

QX Tower Inlet				QX Tower Outlet		
Can ID	721	176	a378	321	2045	793
Location	QX Inlet	QX Inlet	QX Inlet	QX Outlet	QX Outlet	QX Outlet
Run Number	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Date	4/30/2018	5/1/2018	5/1/2018	4/30/2018	5/1/2018	5/1/2018

**Data Descriptors**

- Contamination present. Quantitation suspect.
- Compound coelution problem. Quantitation not reliable.
- Above calibration range. Estimated value based on highest calibration point.
- Compounds below 3 x MDL are reported as '< MQL'
- Reported values are blank corrected

Target Compounds	Conc. ppbv	Conc. ppbv	Conc. ppbv	Conc. ppbv	Conc. ppbv	Conc. ppbv
Propylene	83.76	82.14	25.62	72.09	76.45	20.43
Propane	51.55	55.36	33.08	45.49	50.09	25.20
Dichlorodifluoromethane	0.28	0.31	0.39	0.29	0.32	0.30
Chloromethane	2.74	2.41	0.51	2.74	3.93	0.43
Isobutane	94.75	91.61	12.21	109.26	44.87	13.30
Dichlorotetrafluoroethane	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Vinyl Chloride	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
1-Butene	56.85	53.77	21.66	50.36	62.84	20.08
1,3-Butadiene	3.01	4.41	< MQL	2.83	4.22	< MQL
Butane	1.57	2.54	2.74	1.54	2.23	1.57
trans-2-butene	6.86	6.33	< MQL	5.94	6.64	< MQL
Bromomethane	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
cis-2-butene	3.54	2.73	< MQL	2.55	2.82	< MQL
Chloroethane	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Ethanol	106.39	66.12	18.04	75.57	58.78	16.64
Vinyl Bromide	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Acetonitrile	1.64	2.26	< MQL	2.34	7.85	< MQL



Can ID	721	176	a378	321	2045	793
Location	QX Inlet	QX Inlet	QX Inlet	QX Outlet	QX Outlet	QX Outlet
Run Number	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Date	4/30/2018	5/1/2018	5/1/2018	4/30/2018	5/1/2018	5/1/2018
Target Compounds	Conc. ppbv	Conc. ppbv	Conc. ppbv	Conc. ppbv	Conc. ppbv	Conc. ppbv
Acrolein	169.37	149.40	22.33	143.40	161.90	25.00
Acetone	>442.145	>402.705	101.48	>389.992	>384.943	83.10
iso-Pentane	5.15	18.76	6.72	20.11	6.69	1.79
Trichlorofluoromethane	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Isopropyl Alcohol	64.49	52.81	31.16	35.41	40.06	19.38
1-Pentene	1.08	1.61	0.96	1.97	1.44	< MQL
Acrylonitrile	5.96	< MQL	0.72	5.14	< MQL	< MQL
n-Pentane	0.26	0.72	1.32	0.35	0.31	< MQL
Isoprene	2.03	1.89	0.66	2.04	2.29	< MQL
trans-2-pentene	1.58	1.52	0.69	1.25	1.43	< MQL
cis-2-pentene	2.05	1.78	0.66	1.40	1.26	< MQL
Tert-Butanol	23.59	20.72	1.00	12.03	13.35	0.61
1,1-Dichloroethene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Methylene Chloride	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
3-Chloro-1-Propene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
1,1,2-Trichloro-1,2,2-trifluoroethane	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Carbon Disulfide	< MQL	< MQL	< MQL	0.53	0.84	< MQL
2,2-Dimethylbutane	< MQL	< MQL	0.70	< MQL	< MQL	< MQL
trans-1,2-Dichloroethene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Cyclopentane	< MQL	< MQL	0.77	1.68	30.29	< MQL
2,3-Dimethylbutane	< MQL	< MQL	0.57	< MQL	< MQL	< MQL
1,1-Dichloroethane	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Methyl-t-Butyl-Ether	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Vinyl Acetate	43.93	41.08	3.13	48.62	39.36	3.24
2-Methylpentane	< MQL	< MQL	1.10	< MQL	< MQL	< MQL
2-Butanone	140.46	138.84	11.78	121.79	157.59	8.05
3-Methylpentane	16.02	16.07	1.57	12.65	14.44	0.35
2-Chloroprene	< MQL	0.71	0.16	< MQL	< MQL	< MQL
1-Hexene	4.30	3.93	0.89	3.92	4.52	< MQL
cis-1,2-Dichloroethene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Diisopropyl ether	0.27	< MQL	< MQL	0.28	< MQL	< MQL
Ethyl Acetate	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
n-Hexane	< MQL	0.52	0.85	0.32	0.39	< MQL
Chloroform	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Tetrahydrofuran	1.09	1.73	2.82	< MQL	< MQL	1.93
Ethyl Tert-Butyl Ether	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Methylcyclopentane	< MQL	0.33	0.90	0.40	0.28	< MQL
1,2-Dichloroethane	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL

Can ID	721	176	a378	321	2045	793
Location	QX Inlet	QX Inlet	QX Inlet	QX Outlet	QX Outlet	QX Outlet
Run Number	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Date	4/30/2018	5/1/2018	5/1/2018	4/30/2018	5/1/2018	5/1/2018
Target Compounds	Conc.	Conc.	Conc.	Conc.	Conc.	Conc.
	ppbv	ppbv	ppbv	ppbv	ppbv	ppbv
2,4-Dimethylpentane	0.53	0.68	1.71	0.45	0.49	0.65
1,1,1-Trichloroethane	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Benzene	0.98	1.06	1.05	2.19	2.20	< MQL
Carbon Tetrachloride	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Cyclohexane	2.15	0.38	1.29	1.67	0.93	0.41
2-Methylhexane	0.77	2.40	1.23	2.03	0.38	< MQL
2,3-Dimethylpentane	< MQL	< MQL	0.64	< MQL	< MQL	< MQL
Tert Amyl Methyl Ether	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
3-methylhexane	0.94	1.34	0.92	< MQL	< MQL	0.50
1,2-Dichloropropane	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Bromodichloromethane	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
1,4-Dioxane	51.40	41.04	19.07	37.80	35.69	17.12
Trichloroethene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Isooctane	2.72	2.55	1.06	2.40	2.51	0.34
Methyl Methacrylate	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Heptane	3.93	4.16	1.12	3.48	4.04	< MQL
cis-1,3-Dichloropropene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
4-Methy-2-Pentanone	38.54	34.44	45.11	42.78	44.52	36.41
Methylcyclohexane	0.61	0.65	1.10	0.65	0.62	0.64
trans-1,3-Dichloropropene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
1,1,2-Trichloroethane	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
2,3,4-Trimethylpentane	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Toluene	9.41	10.11	5.77	8.31	9.57	4.63
2-Methylheptane	18.88	18.26	1.01	14.02	14.73	< MQL
Dibromochloromethane	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
3-Methylheptane	< MQL	6.36	< MQL	1.88	2.48	< MQL
1,2-Dibromoethane	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Octane	< MQL	0.85	0.81	< MQL	< MQL	< MQL
Tetrachloroethene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
1,1,1,2-Tetrachloroethane	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Chlorobenzene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Ethylbenzene	< MQL	< MQL	0.65	< MQL	< MQL	< MQL
m-Xylene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
p-Xylene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Bromoform	< MQL	< MQL	0.18	< MQL	< MQL	< MQL
Styrene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
1,1,2,2-Tetrachloroethane	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
o-Xylene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL

Can ID	721	176	a378	321	2045	793
Location	QX Inlet	QX Inlet	QX Inlet	QX Outlet	QX Outlet	QX Outlet
Run Number	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Date	4/30/2018	5/1/2018	5/1/2018	4/30/2018	5/1/2018	5/1/2018
Target Compounds	Conc. ppbv	Conc. ppbv	Conc. ppbv	Conc. ppbv	Conc. ppbv	Conc. ppbv
Nonane	0.87	0.98	0.82	0.82	0.89	< MQL
Cumene	< MQL	0.20	0.70	0.20	0.22	< MQL
n-Propylbenzene	< MQL	0.47	0.63	0.38	0.38	< MQL
m-Ethyltoluene	< MQL	0.51	0.47	0.48	0.54	< MQL
1,3,5-Trimethylbenzene	< MQL	< MQL	0.55	< MQL	< MQL	< MQL
1,2,4-Trimethylbenzene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Tert-Butyl Benzene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
1-Ethyl-4-Methyl Benzene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
o-Ethyltoluene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
1,3-Dichlorobenzene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
1,4-Dichlorobenzene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
n-Decane	< MQL	< MQL	0.50	0.50	< MQL	< MQL
Sec-Butyl Benzene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
1,2,3-Trimethylbenzene	< MQL	0.41	0.45	0.39	< MQL	< MQL
1,2-Dichlorobenzene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
o-Cymene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
1,3-Diethylbenzene	< MQL	0.40	0.57	0.38	0.38	< MQL
1,2-Diethylbenzene	< MQL	0.40	0.61	0.51	0.46	< MQL
n-Butyl Benzene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL
Undecane	1.73	2.68	1.47	2.47	2.27	< MQL
1,2,4-Trichlorobenzene	< MQL	< MQL	0.60	< MQL	0.73	< MQL
Naphthalene	< MQL	< MQL	< MQL	0.58	0.90	< MQL
Dodecane	2.62	34.92	1.55	9.73	42.10	38.70
Hexachlorobutadiene	< MQL	< MQL	< MQL	< MQL	< MQL	< MQL

**Table 7.** Measured concentrations of TO-15 (plus PAMS) compounds in ambient samples and a single field blank.

	Ambient			Field Blank
Can ID	005	794	rk9	709
Sample Type	Ambient	Ambient	Ambient	Field Blank
Location	(lower roof)	(upper roof)	(inside fac.)	
Date	5/1/2018	4/27/2018	5/1/2018	4/30/2018

**Data Descriptors**

- Contamination present. Quantitation suspect.
- Compound coelution problem. Quantitation not reliable.
- Above calibration range. Estimated value based on highest calibration point.
- Compounds below 3 x MDL are reported as '< MQL'
- Reported values are blank corrected

Target Compounds	Conc. ppbv	Conc. ppbv	Conc. ppbv	Conc. ppbv
Propylene	0.46	< MQL	2.30	0.14
Propane	1.49	2.40	10.04	0.30
Dichlorodifluoromethane	0.34	0.31	0.32	< MQL
Chloromethane	0.53	< MQL	0.55	< MQL
Isobutane	0.88	< MQL	1.48	< MQL
Dichlorotetrafluoroethane	< MQL	< MQL	< MQL	< MQL
Vinyl Chloride	< MQL	< MQL	< MQL	< MQL
1-Butene	< MQL	< MQL	< MQL	< MQL
1,3-Butadiene	< MQL	< MQL	< MQL	< MQL
Butane	0.60	< MQL	0.60	< MQL
trans-2-butene	< MQL	< MQL	< MQL	< MQL
Bromomethane	< MQL	< MQL	< MQL	< MQL
cis-2-butene	< MQL	< MQL	< MQL	< MQL
Chloroethane	< MQL	< MQL	< MQL	< MQL
Ethanol	2.70	5.43	14.92	< MQL
Vinyl Bromide	< MQL	< MQL	< MQL	< MQL
Acetonitrile	< MQL	< MQL	0.48	< MQL

Can ID	005	794	rk9	709
Sample Type	Ambient	Ambient	Ambient	Field Blank
Location	(lower roof)	(upper roof)	(inside fac.)	
Date	5/1/2018	4/27/2018	5/1/2018	4/30/2018
Target Compounds	Conc. ppbv	Conc. ppbv	Conc. ppbv	Conc. ppbv
Acrolein	0.77	< MQL	1.74	0.12
Acetone	5.83	< MQL	6.50	< MQL
iso-Pentane	0.82	< MQL	0.66	< MQL
Trichlorofluoromethane	< MQL	< MQL	0.21	< MQL
Isopropyl Alcohol	5.57	3.10	9.80	< MQL
1-Pentene	< MQL	< MQL	< MQL	< MQL
Acrylonitrile	1.19	< MQL	< MQL	< MQL
n-Pentane	15.14	< MQL	0.13	< MQL
Isoprene	< MQL	< MQL	0.57	< MQL
trans-2-pentene	< MQL	< MQL	< MQL	< MQL
cis-2-pentene	< MQL	< MQL	< MQL	< MQL
Tert-Butanol	< MQL	< MQL	0.15	< MQL
1,1-Dichloroethene	< MQL	< MQL	< MQL	< MQL
Methylene Chloride	< MQL	< MQL	< MQL	< MQL
3-Chloro-1-Propene	< MQL	< MQL	< MQL	< MQL
1,1,2-Trichloro-1,2,2-trifluoroethane	< MQL	< MQL	< MQL	< MQL
Carbon Disulfide	< MQL	< MQL	< MQL	< MQL
2,2-Dimethylbutane	< MQL	< MQL	< MQL	< MQL
trans-1,2-Dichloroethene	< MQL	< MQL	< MQL	< MQL
Cyclopentane	< MQL	< MQL	< MQL	< MQL
2,3-Dimethylbutane	< MQL	< MQL	< MQL	< MQL
1,1-Dichloroethane	< MQL	< MQL	< MQL	< MQL
Methyl-t-Butyl-Ether	< MQL	< MQL	< MQL	< MQL
Vinyl Acetate	< MQL	< MQL	0.73	< MQL
2-Methylpentane	< MQL	< MQL	< MQL	< MQL
2-Butanone	< MQL	< MQL	18.27	< MQL
3-Methylpentane	< MQL	< MQL	0.38	< MQL
2-Chloroprene	< MQL	< MQL	< MQL	< MQL
1-Hexene	< MQL	< MQL	< MQL	< MQL
cis-1,2-Dichloroethene	< MQL	< MQL	< MQL	< MQL
Diisopropyl ether	< MQL	< MQL	< MQL	< MQL
Ethyl Acetate	< MQL	< MQL	< MQL	< MQL
n-Hexane	< MQL	< MQL	0.36	< MQL
Chloroform	< MQL	< MQL	< MQL	< MQL
Tetrahydrofuran	< MQL	< MQL	< MQL	< MQL
Ethyl Tert-Butyl Ether	< MQL	< MQL	< MQL	< MQL
Methylcyclopentane	< MQL	< MQL	0.15	< MQL
1,2-Dichloroethane	< MQL	< MQL	< MQL	< MQL

Can ID	005	794	rk9	709
Sample Type	Ambient	Ambient	Ambient	Field Blank
Location	(lower roof)	(upper roof)	(inside fac.)	
Date	5/1/2018	4/27/2018	5/1/2018	4/30/2018
Target Compounds	Conc.	Conc.	Conc.	Conc.
	ppbv	ppbv	ppbv	ppbv
2,4-Dimethylpentane	< MQL	< MQL	0.09	< MQL
1,1,1-Trichloroethane	< MQL	< MQL	< MQL	< MQL
Benzene	< MQL	< MQL	< MQL	< MQL
Carbon Tetrachloride	< MQL	< MQL	< MQL	< MQL
Cyclohexane	0.25	< MQL	0.26	< MQL
2-Methylhexane	< MQL	< MQL	0.52	< MQL
2,3-Dimethylpentane	< MQL	< MQL	< MQL	< MQL
Tert Amyl Methyl Ether	< MQL	< MQL	< MQL	< MQL
3-methylhexane	< MQL	< MQL	0.63	< MQL
1,2-Dichloropropane	< MQL	< MQL	< MQL	< MQL
Bromodichloromethane	< MQL	< MQL	< MQL	< MQL
1,4-Dioxane	< MQL	< MQL	< MQL	< MQL
Trichloroethene	< MQL	< MQL	< MQL	< MQL
Isooctane	< MQL	< MQL	< MQL	< MQL
Methyl Methacrylate	< MQL	< MQL	< MQL	< MQL
Heptane	< MQL	< MQL	0.84	< MQL
cis-1,3-Dichloropropene	< MQL	< MQL	< MQL	< MQL
4-Methy-2-Pentanone	< MQL	< MQL	< MQL	< MQL
Methylcyclohexane	< MQL	< MQL	0.95	< MQL
trans-1,3-Dichloropropene	< MQL	< MQL	< MQL	< MQL
1,1,2-Trichloroethane	< MQL	< MQL	< MQL	< MQL
2,3,4-Trimethylpentane	< MQL	< MQL	< MQL	< MQL
Toluene	1.22	< MQL	33.75	< MQL
2-Methylheptane	< MQL	< MQL	0.25	< MQL
Dibromochloromethane	< MQL	< MQL	< MQL	< MQL
3-Methylheptane	< MQL	< MQL	< MQL	< MQL
1,2-Dibromoethane	< MQL	< MQL	< MQL	< MQL
Octane	< MQL	< MQL	< MQL	< MQL
Tetrachloroethene	< MQL	< MQL	< MQL	< MQL
1,1,1,2-Tetrachloroethane	< MQL	< MQL	< MQL	< MQL
Chlorobenzene	< MQL	< MQL	< MQL	< MQL
Ethylbenzene	< MQL	< MQL	< MQL	< MQL
m-Xylene	< MQL	< MQL	< MQL	< MQL
p-Xylene	< MQL	< MQL	< MQL	< MQL
Bromoform	< MQL	< MQL	< MQL	< MQL
Styrene	< MQL	< MQL	< MQL	< MQL
1,1,2,2-Tetrachloroethane	< MQL	< MQL	< MQL	< MQL
o-Xylene	< MQL	< MQL	< MQL	< MQL

	Can ID	005	794	rk9	709
	Sample Type	Ambient	Ambient	Ambient	Field Blank
	Location	(lower roof)	(upper roof)	(inside fac.)	
	Date	5/1/2018	4/27/2018	5/1/2018	4/30/2018
Target Compounds		Conc. ppbv	Conc. ppbv	Conc. ppbv	Conc. ppbv
Nonane		< MQL	< MQL	< MQL	< MQL
Cumene		< MQL	< MQL	0.08	< MQL
n-Propylbenzene		< MQL	< MQL	< MQL	< MQL
m-Ethyltoluene		< MQL	< MQL	< MQL	< MQL
1,3,5-Trimethylbenzene		< MQL	< MQL	< MQL	< MQL
1,2,4-Trimethylbenzene		< MQL	< MQL	< MQL	< MQL
Tert-Butyl Benzene		< MQL	< MQL	< MQL	< MQL
1-Ethyl-4-Methyl Benzene		< MQL	< MQL	< MQL	< MQL
o-Ethyltoluene		< MQL	< MQL	< MQL	< MQL
1,3-Dichlorobenzene		< MQL	< MQL	< MQL	< MQL
1,4-Dichlorobenzene		< MQL	< MQL	< MQL	< MQL
n-Decane		< MQL	< MQL	< MQL	< MQL
Sec-Butyl Benzene		< MQL	< MQL	< MQL	< MQL
1,2,3-Trimethylbenzene		< MQL	< MQL	< MQL	< MQL
1,2-Dichlorobenzene		< MQL	< MQL	< MQL	< MQL
o-Cymene		< MQL	0.54	< MQL	< MQL
1,3-Diethylbenzene		< MQL	< MQL	< MQL	< MQL
1,2-Diethylbenzene		< MQL	< MQL	< MQL	< MQL
n-Butyl Benzene		< MQL	< MQL	< MQL	< MQL
Undecane		0.63	< MQL	0.73	< MQL
1,2,4-Trichlorobenzene		< MQL	< MQL	< MQL	< MQL
Naphthalene		< MQL	< MQL	0.35	< MQL
Dodecane		6.48	1.31	2.87	0.17
Hexachlorobutadiene		< MQL	< MQL	< MQL	< MQL